

(19)



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Office européen des brevets



(11)

EP 0 757 080 A2

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:
05.02.1997 Bulletin 1997/06

(51) Int Cl.⁶: **C08L 83/04, C09J 183/04,
H01L 21/50**

(21) Application number: **96305624.7**

(22) Date of filing: **31.07.1996**

(84) Designated Contracting States:
DE FR GB

(30) Priority: **04.08.1995 JP 219859/95
04.08.1995 JP 219860/95**

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(54) Curable organosiloxane compositions and semiconductor devices

(57) A curable organosiloxane composition useable as a silicone die attach adhesive, an electrically conductive silicone rubber composition each comprising a composition that cures through both addition and condensation reactions, that will not impair wire bondability to a semiconductor chip or lead frame after the semicon-

ductor chip has been bonded to a substrate or package; and semiconductor devices in which the semiconductor chip is bonded to its substrate or package using the curable organosiloxane composition and in which the semiconductor device evidences high reliability by virtue of the use of the curable organosiloxane composition.

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Description

This invention relates to curable organosiloxane compositions and semiconductor devices. More particularly, the present invention provides curable organosiloxane compositions that are usable as silicone die attach adhesives which adhere a semiconductor device to a substrate without impairment of the bondability of a wire to a semiconductor chip or to a lead frame. The invention also introduces electrically conductive silicone rubber compositions which yield a highly electrically conductive silicone rubber upon cure that is not accompanied by contamination of the area around the composition with a low molecular-weight silicone oil. The invention also produces semiconductor devices that evidence a high reliability by virtue of the use of the silicone die attach adhesive or the electrically conductive silicone rubber composition.

Semiconductor devices are fabricated by first bonding the semiconductor chip to a substrate using a solder, such as the Au/Si eutectic, or by using an epoxy, polyimide, or silicone die attach adhesive. The substrate is often referred to as a package or a semiconductor package. The semiconductor chip and lead frame are then electrically connected in a wire bonding operation using gold or aluminum wire. The semiconductor chip is thereafter resin-sealed using an epoxy resin or a polyphenylene sulfide resin. Silicone die attach adhesives are used because they are able to relax the stresses induced in the semiconductor chip. However, the known silicone die attach adhesives are associated with such problems as an impairment in the wire bondability (wire joinability) of the semiconductor chip to the lead frame; defective adherence between the sealing resin and the semiconductor chip, substrate or lead frame; and an impaired moisture resistance by the semiconductor device.

To solve these problems, 3-157474 proposed a silicone die attach adhesive comprising an addition-curing organopolysiloxane composition with a reduced content of volatile low-molecular-weight siloxane. However, even this silicone die attach adhesive cannot totally overcome the problems of impairment of the wire bondability of the semiconductor chip and lead frame, and the degeneration in moisture resistance by the semiconductor device.

As a result of various investigations, it has been confirmed that during attachment of the semiconductor chip to a substrate using silicone die attach adhesives of the prior art, the surface of the semiconductor chip, substrate, lead frame, etc., will become contaminated by low molecular-weight silicone oil that bleeds out of the silicone die attach adhesive prior to its thermosetting. It was also confirmed that this contamination resulted in the impairment of wire bondability to these surfaces, defective bonding with the sealant resin, and impaired moisture resistance of the semiconductor device.

First, we used an addition-curing silicone die attach adhesive that cured immediately at room temperature to inhibit this silicone oil bleed from the adhesive. However, the handling properties of this silicone die attach adhesive were so severely compromised that the die bonding operation could not be performed.

As a result of extensive investigations directed to solving the problems described above, we then found that these problems are solved by attaching the semiconductor chip to its substrate with a curable organopolysiloxane composition that cures by both addition and condensation reactions. With the use of such a composition, a cured film is immediately formed - through the action of atmospheric moisture - on the surface of the silicone die attach adhesive. Our present invention is based on this discovery.

Conductive silicone rubber compositions cure to give silicone rubbers that evidence an excellent conductivity. These compositions are used, for example, as adhesives to fix quartz plates on a substrate, e.g.; quartz filters or quartz resonators mounted in quartz oscillators, as adhesives for fixing piezoelectric substrates in packages; as shielding materials from electromagnetic radiation; and as die attach adhesives for bonding semiconductor chips on a substrate or package. However, the known conductive silicone rubber compositions are also associated with such problems as described above for silicone die attach adhesives.

In order to solve these problems, JP-A 3-170581 suggested an addition-curing conductive silicone rubber composition with a reduced content of volatile low molecular-weight siloxane. However, even this conductive silicone rubber composition cannot provide an acceptable inhibition of the impairment in wire bondability and the degeneration in moisture resistance by the semiconductor device.

As a result of further investigations, we confirmed that during the attachment of a semiconductor chip to a substrate or package using conductive silicone rubber compositions of the prior art, the surface of the semiconductor chip, substrate, package, lead frame, etc., will become contaminated by low molecular-weight silicone oil bleeds from the composition prior to its thermosetting. It was also determined that this contamination resulted in the impairment of wire bondability to these surfaces, defective bonding with the sealant resin, and impaired moisture resistance of the semiconductor device.

Again, we attempted the use of an addition-curing conductive silicone rubber composition that cured immediately at room temperature to inhibit this bleed of low molecular-weight silicone oil. However, this composition had severely impaired handling properties.

As a result of more investigations, we found that these additional problems are solved by attaching the semiconductor chip to its substrate or package with a conductive silicone rubber composition that cures by both addition and

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problem

condensation reactions. With the use of such a composition, a cured film is immediately formed - through the action of atmospheric moisture - on the surface of the applied composition. This action prevents contamination of the area around the composition by low molecular-weight silicone oil. The present invention is also based on this discovery.

The significant objects of the present invention are: 1) to provide a silicone die attach adhesive that will not impair wire bondability to the semiconductor chip or lead frame after the semiconductor chip has been bonded to its substrate; 2) to provide a conductive silicone rubber composition whose cure yields a highly conductive silicone rubber that is not accompanied by contamination of the area around the composition with low molecular-weight silicone oil; and 3) to provide a semiconductor device that evidences a high reliability because the semiconductor chip contained therein is bonded to its substrate or package by the use of either a silicone die attach adhesive or an electrically conductive silicone rubber composition.

In a first embodiment, the present invention relates to a silicone die attach adhesive comprising a curable organopolysiloxane composition that cures through both an addition and a condensation reaction. The invention also relates to a similar electrically conductive silicone rubber composition. Finally, this invention provides semiconductor devices in which the semiconductor is bonded to its substrate or package by the use of either the electrically above conductive silicone rubber composition or the above silicone die attach adhesive.

The silicone die attach adhesive of the present invention comprises a curable organopolysiloxane composition that cures by both addition and condensation reactions. An example of such a composition comprises

(A) 100 parts by weight of a mixture of

(a) 5 to 95 weight% of an alkoxy-substituted organopolysiloxane that has a viscosity at 25°C of 20 to 200,000 mPa.s, that contains an average of at least two silicon-bonded alkoxy groups per molecule, and that does not contain any silicon-bonded alkenyl groups; and

(b) 95 to 5 weight% of an alkenyl-substituted organopolysiloxane that has a viscosity at 25°C of 20 to 200,000 mPa.s, that contains an average of at least two silicon-bonded alkenyl groups per molecule, and that does not contain any silicon-bonded alkoxy groups;

(B) an organopolysiloxane that has a viscosity at 25°C of 2 to 20,000 mPa.s and that contains an average of at least two silicon-bonded hydrogen atoms per molecule, in a quantity that affords a value from 0.3 to 20 for the molar ratio of silicon-bonded hydrogen atoms in component (B) to silicon-bonded alkenyl groups in component (b);
(C) 0.01 to 10 parts by weight of a condensation reaction catalyst; and
(D) a catalytic quantity of platinum catalyst.

Another aspect of the present invention is a curable conductive silicone rubber composition comprising

(A') 100 parts by weight of an alkenyl-substituted organopolysiloxane that has a viscosity at 25°C of 20 to 200,000 mPa.s, that contains an average of at least two silicon-bonded alkenyl groups per molecule, and that optionally contains an average of at least one silicon-bonded alkoxy group per molecule;

(B') an organopolysiloxane that has a viscosity at 25°C of 2 to 20,000 mPa.s, that contains an average of at least 2 silicon-bonded hydrogen atoms per molecule, and that optionally contains an average of at least one silicon-bonded alkoxy group per molecule, in a quantity that affords a value from 0.3 to 20 for the molar ratio of silicon-bonded hydrogen atoms in component (B') to silicon-bonded alkenyl groups in component (A');
(C) 0.01 to 10 parts by weight of a condensation reaction catalyst; and
(D) a catalytic quantity of platinum catalyst;

with the proviso that at least one of components (A') and (B') contains an average of at least two silicon-bonded alkoxy groups per molecule.

Component (A) is a mixture of (a) an alkoxy-substituted organopolysiloxane that has a viscosity at 25°C of 20 to 200,000 mPa.s, that contains an average of at least two silicon-bonded alkoxy groups per molecule, and that does not contain any silicon-bonded alkenyl groups; and (b) an alkenyl-substituted organopolysiloxane that has a viscosity at 25°C of 20 to 200,000 mPa.s, that contains an average of at least two silicon-bonded alkenyl groups per molecule, and that does not contain any silicon-bonded alkoxy groups. Impaired wire bondability to the semiconductor chip and lead frame or impaired moisture resistance of the semiconductor device will occur when component (a) contains an average of less than two silicon-bonded alkoxy groups per molecule. The molecular structure of this component is exemplified by straight chain, partially branched straight chain, and branched chain, wherein straight chain is preferred. The silicon-bonded alkoxy in component (a) is exemplified by methoxy, ethoxy, propoxy, butoxy, methoxymethoxy, and methoxyethoxy, with methoxy being preferred. The alkoxy group may be bonded in the terminal or nonterminal position on the molecular chain, but bonding at the molecular chain terminals is preferred for the corresponding good reactivity.

The alkoxy may also be directly bonded to main-chain silicon, or it may be bonded to a silicon atom that in turn is bonded to main-chain silicon across an alkylene group. The non-alkoxy silicon-bonded groups in component (a) are exemplified by alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl; cycloalkyl groups such as cyclopentyl or cyclohexyl; aryl groups such as phenyl, tolyl, xylyl, and naphthyl; aralkyl groups such as benzyl, phenethyl, and phenylpropyl; and haloalkyl groups such as 3-chloropropyl, or 3,3,3-trifluoropropyl. The preferred non-alkoxy silicon-bonded groups are methyl and phenyl. The viscosity of component (a) at 25°C is from 20 to 200,000 mPa.s and is preferably from 100 to 100,000 mPa.s. A viscosity at 25°C below 20 mPa.s leads to a decline in the physical properties of the ultimately obtained cured product, for example, in the flexibility or elongation. Viscosities in excess of 200,000 mPa.s lead to a deterioration in the handling characteristics of the corresponding composition.

Component (a) is exemplified by the following:

trimethoxysiloxy-endblocked dimethylpolysiloxanes,
 trimethoxysiloxy-endblocked dimethylsiloxane-methylphenylsiloxane copolymers,
 triethoxysiloxy-endblocked dimethylpolysiloxanes,
 tripropoxysiloxy-endblocked dimethylpolysiloxanes,
 methyltrimethoxysiloxy-endblocked dimethylpolysiloxanes,
 methyltriethoxysiloxy-endblocked dimethylpolysiloxanes,
 methyltrimethoxysiloxy-endblocked dimethylsiloxane-methylphenylsiloxane copolymers,
 trimethoxysilylethyltrimethylsiloxy-endblocked dimethylpolysiloxanes,
 triethoxysilylethyltrimethylsiloxy-endblocked dimethylpolysiloxanes,
 trimethoxysilylpropyltrimethylsiloxy-endblocked dimethylpolysiloxanes,
 trimethoxysilylethyltrimethylsiloxy-endblocked dimethylsiloxane-methylphenylsiloxane copolymers,
 methyltrimethoxysilylethyltrimethylsiloxy-endblocked dimethylpolysiloxanes,
 methyltrimethoxysilylethyltrimethylsiloxy-endblocked dimethylsiloxane-methylphenylsiloxane copolymers,
 trimethylsiloxy-endblocked dimethylsiloxanemethyl(trimethoxysilylethyl)siloxane copolymers,
 trimethylsiloxy-endblocked dimethylsiloxanemethyl(triethoxysilylethyl)siloxane copolymers, and
 trimethoxysilylethyltrimethylsiloxy-endblocked dimethylsiloxanemethyl(trimethylsilylethyl)siloxane copolymers.

Component (a) may consist of a single such alkoxy-substituted organopolysiloxane or any combination thereof

The resulting composition will also not evidence an acceptable cure when component (b) contains an average of less than two silicon-bonded alkenyl groups per molecule. The molecular structure of this component is exemplified by straight chain, partially branched straight chain, branched, cyclic, and resin structures. The silicon-bonded alkenyl in component (b) is exemplified by vinyl, allyl, butenyl, pentenyl, and hexenyl, wherein vinyl is preferred. The alkenyl can be bonded at terminal or nonterminal positions on the molecular chain, but bonding at the molecular chain terminals is preferred. The non-alkenyl silicon-bonded groups of component (b) are exemplified by alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl; cycloalkyl groups such as cyclopentyl, or cyclohexyl; aryl groups such as phenyl, tolyl, xylyl, and naphthyl; aralkyl groups such as benzyl, phenethyl, and phenylpropyl; and haloalkyl groups such as 3-chloropropyl, or 3,3,3-trifluoropropyl. The preferred non-alkoxy silicon-bonded groups are methyl and phenyl. The viscosity of component (b) at 25°C is from 20 to 200,000 mPa.s and is preferably from 100 to 100,000 mPa.s. A viscosity at 25°C below 20 mPa.s leads to a decline in the physical properties of the ultimately obtained cured product. Viscosities in excess of 200,000 mPa.s lead to a deterioration in the handling characteristics of the corresponding composition.

Component (b) is exemplified by

trimethylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers;
 trimethylsiloxy-endblocked methylvinylpolysiloxanes;
 trimethylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methylphenylsiloxane copolymers;
 dimethylvinylsiloxy-endblocked dimethylpolysiloxanes;
 dimethylvinylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers;
 dimethylvinylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methylphenylsiloxane copolymers;
 organopolysiloxanes composed of $(\text{CH}_2=\text{CH})(\text{CH}_3)_2\text{SiO}_{1/2}$ and $\text{SiO}_{4/2}$ units;
 organopolysiloxanes composed of $(\text{CH}_3)_3\text{SiO}_{1/2}$, $(\text{CH}_2=\text{CH})(\text{CH}_3)_2\text{SiO}_{1/2}$, and $\text{SiO}_{4/2}$ units; and
 organopolysiloxanes composed of $(\text{CH}_3)_3\text{SiO}_{1/2}$, $(\text{CH}_2=\text{CH})(\text{CH}_3)_2\text{SiO}_{1/2}$, $(\text{CH}_3)_2\text{SiO}_{2/2}$, and $\text{SiO}_{4/2}$ units.

Component (b) may consist of a single such organopolysiloxane or any combination thereof.

Component (a) constitutes from 5 to 95 weight% of component (A) with component (b) making up the balance. The presence of less than 5 weight% of component (a) in component (A) leads to an impairment in the wire bondability to the semiconductor chip and lead frame, or an impairment of the moisture resistance of the semiconductor device.

The presence of more than 95 weight% causes the resulting composition to have a slow cure.

The organopolysiloxane (B) undergoes an addition reaction with component (b) and thereby crosslinks component (b). Component (B) must contain an average of at least two silicon-bonded hydrogen atoms per molecule. The resulting composition will suffer from such problems as an unsatisfactory cure or a slow cure when component (B) contains an average of less than two silicon-bonded hydrogen atoms per molecule. The molecular structure of component (B) is straight chain, partially branched straight chain, cyclic, and resin structures. The silicon-bonded hydrogen may be bonded in terminal or nonterminal position on the molecular chain. The silicon-bonded groups in component (B) other than hydrogen are exemplified by alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl; cycloalkyl groups such as cyclopentyl or cyclohexyl; aryl groups such as phenyl, tolyl, naphthyl and xyl; aralkyl groups such as benzyl, phenethyl, and phenylpropyl; and haloalkyl groups such as 3-chloropropyl, or 3,3,3-trifluoropropyl. Methyl and phenyl are preferred. Other groups that may be present in component (B) on an optional basis are, for example, silicon-bonded alkoxy groups, epoxy-functional monovalent organic groups, and acrylic-functional monovalent organic groups. The silicon-bonded alkoxy groups are exemplified by the same alkoxy groups as above. The epoxy-functional monovalent organic groups are exemplified by oxiranyllalkyl groups such as 4-oxiranylbutoyl or 8-oxiranyloctyl; by glycidoxylalkyl groups such as 3-glycidoxypropyl or 4-glycidoxybutyl; and by 2-(3,4-epoxycyclohexyl)ethyl. The acrylic-functional monovalent organic groups are exemplified by 3-methacryloxypropyl or 4-methacryloxybutyl. Component (B) has a viscosity at 25°C of 2 to 20,000 mPa.s. When component (B) has a viscosity at 25°C below 2 mPa.s, it becomes so volatile that the resulting composition will be unstable. The handling characteristics of the resulting composition are poor when component (B) has a viscosity at 25°C in excess of 20,000 mPa.s.

Component (B) is exemplified by

trimethylsiloxy-endblocked methylhydrogenpolysiloxanes;
trimethylsiloxy-endblocked dimethylsiloxane-methylhydrogensiloxane copolymers;
dimethylhydrogensiloxy-endblocked dimethylpolysiloxanes;
dimethylhydrogensiloxy-endblocked dimethylsiloxane-methylhydrogensiloxane copolymers;
organopolysiloxanes composed of $(\text{CH}_3)_2\text{HSiO}_{1/2}$ and $\text{SiO}_{4/2}$ units;
organopolysiloxanes composed of $(\text{CH}_3)_3\text{SiO}_{1/2}$, $(\text{CH}_3)_2\text{HSiO}_{1/2}$, and $\text{SiO}_{4/2}$ units; and
organopolysiloxanes composed of $(\text{CH}_3)_3\text{SiO}_{1/2}$, $(\text{CH}_3)_2\text{HSiO}_{1/2}$, $(\text{CH}_3)_2\text{SiO}_{2/2}$, and $\text{SiO}_{4/2}$ units.

Examples of the silicon-bonded alkoxy-functional organopolysiloxane are

trimethylsiloxy-endblocked methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane copolymers,
trimethylsiloxy-endblocked dimethylsiloxane-methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane copolymers,
cyclic methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane copolymers, and
cyclic dimethylsiloxane-methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane copolymers.

Examples of organopolysiloxane containing both silicon-bonded alkoxy and epoxy-functionalized monovalent organic groups are

trimethylsiloxy-endblocked methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane-methyl(3-glycidoxypropyl)siloxane copolymers,
trimethylsiloxy-endblocked dimethylsiloxane-methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane-methyl(3-glycidoxypropyl)siloxane copolymers,
cyclic methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane-methyl(3-glycidoxypropyl)siloxane copolymers, and
cyclic dimethylsiloxane-methylhydrogensiloxane-methyl(trimethoxysilylethyl)siloxane-methyl(3-glycidoxypropyl)siloxane copolymers.

Component (B) may consist of a single such organopolysiloxane or any combination thereof.

Component (B) is added in an amount that affords a value from 0.3 to 20 for the molar ratio of silicon-bonded hydrogen atoms in component (B) to silicon-bonded alkenyl groups in component (b). When this molar ratio falls below 0.3, the resulting composition will not manifest an acceptable cure. When this molar ratio exceeds 20, the ultimately obtained cured product will exhibit diminished physical properties.

The condensation reaction catalyst (C) is a catalyst that accelerates the condensation reaction of component (a). Component (C) is exemplified by condensation reaction catalysts of organotitanium, organozirconium and organoaluminum compounds. The organotitanium condensation catalysts are exemplified by organotitanate esters such as tetrabutyl titanate or tetraisopropyl titanate; and by organotitanium chelate compounds such as diisopropoxybis(acetyl-

lacetate)titanium or diisopropoxybis(ethyl acetoacetate)titanium. The organozirconium reaction catalysts are exemplified by organozirconium esters such as zirconium tetrapropylate or zirconium tetrabutylate, and by organozirconium chelate compounds such as zirconium diacetate, zirconium tetra(acetylacetonate), tributoxyzirconium acetylacetonate, dibutoxyzirconium bis(acetylacetonate), tributoxyzirconium acetoacetate, and dibutoxyzirconium acetylacetonate (ethyl acetoacetate). The organoaluminum reaction catalysts are exemplified by organoaluminum esters such as aluminum triethylate, aluminum triisopropylate, aluminum tri(sec-butylate), and mono(sec-butoxy)aluminum diisopropylate; or by organoaluminum chelate compounds such as diisopropoxyaluminum (ethyl acetoacetate), aluminum tris(ethyl acetoacetate), aluminum bis(ethyl acetoacetate) monoacetylacetonate, and aluminum tris(acetylacetonate). These catalysts may be used individually or in combinations of two or more as component (C).

Component (C) is used at 0.01 to 10 weight parts and preferably at 0.1 to 5 weight parts, in each case per 100 weight parts component (A). The use of less than 0.01 weight part of component (C) per 100 weight parts of component (A) leads to the problems of impairment of wire bondability to the semiconductor chip and lead frame, or to impairment of the moisture resistance of the semiconductor device. The use of more than 10 weight parts leads to such problems as an impaired storage stability or impaired handling characteristics for the resulting composition.

The platinum catalyst (D) is a catalyst that accelerates the addition reaction between components (b) and (B), and it is exemplified by platinum black, platinum-on-active carbon, platinum-on-silica micropowder, chloroplatinic acid, alcohol solutions of chloroplatinic acid, platinum-olefin complexes, platinum-vinylsiloxane complexes, and microparticulate catalysts comprising thermoplastic resin containing a platinum catalyst. This thermoplastic resin is exemplified by silicone resins, polycarbonate resins, acrylic resins, nylon resins, and polyester resins. Moreover, the thermoplastic resin preferably has a softening point from 5°C to 200°C and preferably has a particle size from 0.01 to 10 micrometers. Component (D) is added in a catalytic amount. In specific terms, it is preferably added in an amount that provides from 0.01 to 1,000 weight-ppm, and more preferably from 0.5 to 200 weight-ppm, in each case as platinum metal atoms in component (D) based on the total composition.

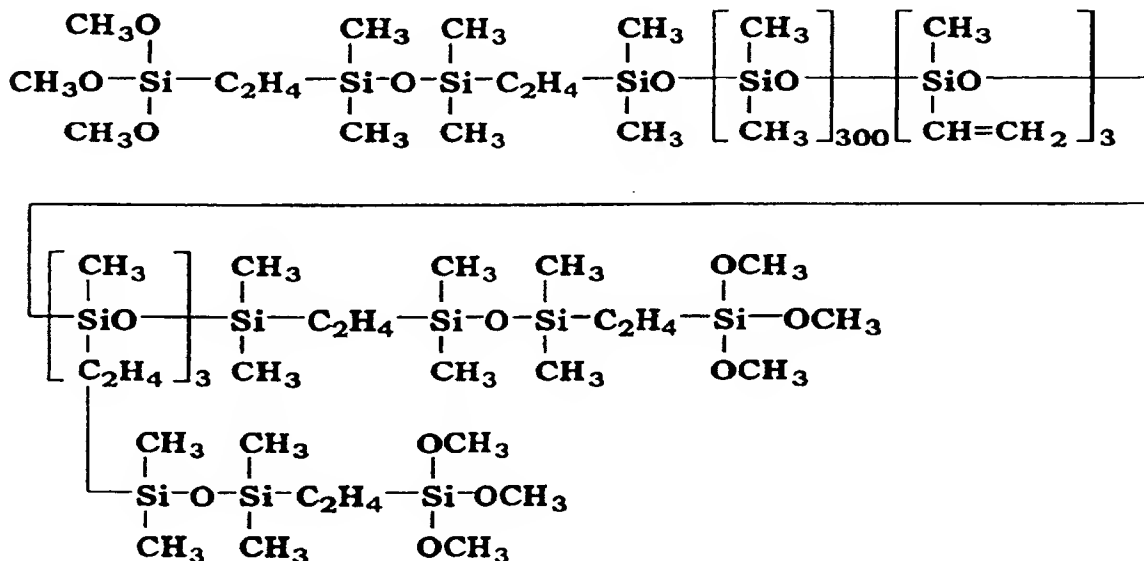
Our silicone die attach adhesive is prepared by mixing components (A) to (D) to homogeneity. However, to improve its adherence, the composition may optionally contain an organosilicon compound that bears silicon-bonded alkoxy, epoxy-functional monovalent organic, and silicon-bonded alkenyl groups. Such a compound is exemplified by 3-glycidoxypropyldimethoxysiloxy-endblocked methylvinylpolysiloxanes, 3-glycidoxypropyldimethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers, 3-glycidoxypropyldiethoxysiloxy-endblocked methylvinylpolysiloxanes, and 3-glycidoxypropyldiethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers. This organosilicon compound is preferably added at from 0.01 to 20 weight parts per 100 weight parts component (A).

In our conductive silicone rubber composition, component (A') is an organopolysiloxane that contains an average of at least two silicon-bonded alkenyl groups per molecule and may or may not contain any silicon-bonded alkoxy groups. The resulting composition will not evidence an acceptable cure when component (A') contains an average of less than two silicon-bonded alkenyl groups per molecule. The molecular structure of component (A') is exemplified by straight chain, partially branched straight chain, and branched chain structures, with straight chain structures being preferred. The silicon-bonded alkenyl in component (A') is exemplified by vinyl, allyl, butenyl, pentenyl, hexenyl, and heptenyl, wherein vinyl is preferred. The alkenyl can be bonded at the terminal or nonterminal position on the molecular chain. The non-alkenyl silicon-bonded groups in component (A') are exemplified by alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl; cycloalkyl groups such as cyclopentyl or cyclohexyl; aryl groups such as phenyl, tolyl, xylyl, and naphthyl; aralkyl groups such as benzyl, phenethyl and phenylpropyl; haloalkyl groups such as 3-chloropropyl or 3,3,3-trifluoropropyl; and alkoxy groups such as methoxy, ethoxy, propoxy, butoxy, methoxymethoxy, and methoxyethoxy. Other groups which may also be present in component (A') on an optional basis are, for example, epoxy-functional monovalent organic groups and acrylic-functional monovalent organic groups. The epoxy-functional monovalent organic groups are exemplified by the same groups as elaborated above, and the acrylic-functional monovalent organic groups are likewise exemplified by the same groups as above. When component (B') contains an average of less than two silicon-bonded alkoxy groups per molecule, component (A') must contain an average of at least 2 silicon-bonded alkoxy groups per molecule. The viscosity of component (A') at 25°C is from 20 to 200,000 mPa.s, and is preferably from 100 to 100,000 mPa.s. A viscosity at 25°C below 20 mPa.s leads to a decline in the physical properties of the ultimately obtained cured product. Viscosities in excess of 200,000 mPa.s lead to a deterioration in the handling characteristics of the corresponding composition.

Component (A') is exemplified by the component (b) organopolysiloxane described above. For the case of organopolysiloxane also containing silicon-bonded alkoxy, component (A') is exemplified as follows:

trimethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers,
 trimethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methylphenylsiloxane copolymers,
 triethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers,
 tripropoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers,
 methyltrimethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers,

methyldiethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers, methyldimethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methylphenylsiloxane copolymers, trimethylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methyl(trimethoxysilylethyl)siloxane copolymers, trimethylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methyl(triethoxysilylethyl)siloxane copolymers, trimethoxysilylethyldimethylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methyl(trimethylsilylethyl)siloxane copolymers, and an organopolysiloxane of the following formula.

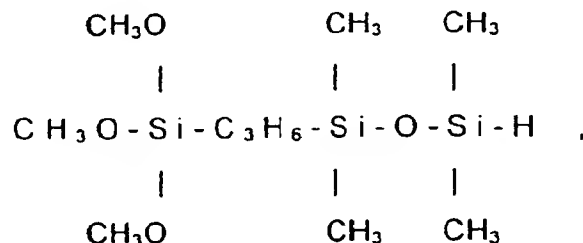
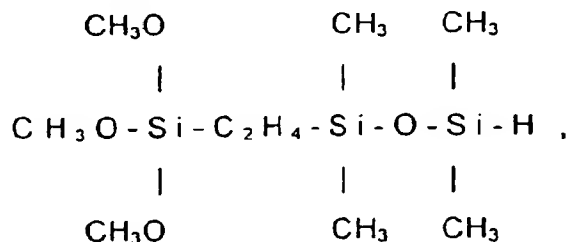


For the case of organopolysiloxane also containing silicon-bonded alkoxy and epoxy-functional monovalent organic groups, component (A') is exemplified as follows:

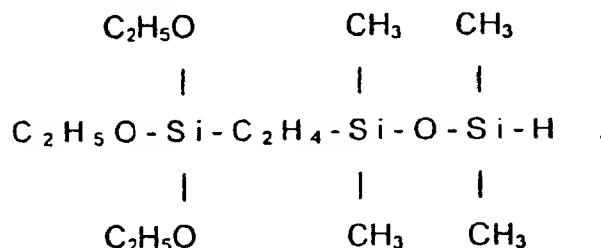
3-glycidoxypropyldimethoxysiloxy-endblocked methylvinylpolysiloxanes, 3-glycidoxypropyldimethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers, 3-glycidoxypropyldiethoxysiloxy-endblocked methylvinylpolysiloxanes, 3-glycidoxypropyldiethoxysiloxy-endblocked dimethylsiloxane-methylvinylsiloxane copolymers, trimethylsiloxy-endblocked methylvinylsiloxane-methyl(trimethoxysilylethyl)siloxane-methyl(3-glycidoxypropyl)siloxane copolymers, and trimethylsiloxy-endblocked dimethylsiloxane-methylvinylsiloxane-methyl(trimethoxysilylethyl)siloxane-methyl(3-glycidoxypropyl)siloxane copolymers.

Component (A') may consist of a single such organopolysiloxane or any combination thereof.

A component (A') that contains an average of at least two silicon-bonded alkoxy groups per molecule can be prepared, for example, by the platinum-catalyzed substoichiometric addition of an organosilicon compound functionalized with both Si-bonded alkoxy and Si-bonded hydrogen to organopolysiloxane containing at least 3 silicon-bonded alkenyl groups in each molecule. It may also be prepared by running a condensation reaction in the presence of a condensation reaction catalyst between organopolysiloxane having in each molecule at least 2 silicon-bonded alkenyl and silanol, and an organosilicon compound having at least 2 silicon-bonded alkoxy in each molecule. The following are examples of the organosilicon compound functionalized with both Si-bonded alkoxy and Si-bonded hydrogen: trimethoxysilane, triethoxysilane, tripropoxysilane, methyldimethoxysilane, and organosilicon compound of the formula



or

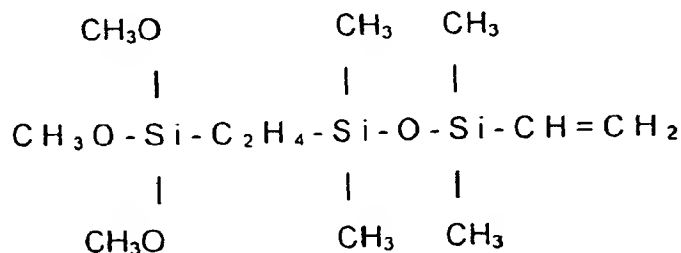


The following are examples of an organosilicon compound having at least 2 silicon-bonded alkoxy groups in each molecule: tetramethoxysilane, tetraethoxysilane, tetrapropoxysilane, methyltrimethoxysilane, ethyltrimethoxysilane, methyltriethoxysilane, hexamethoxydisilylethane, hexamethoxydisilylpropane, and hexamethoxydisiloxane.

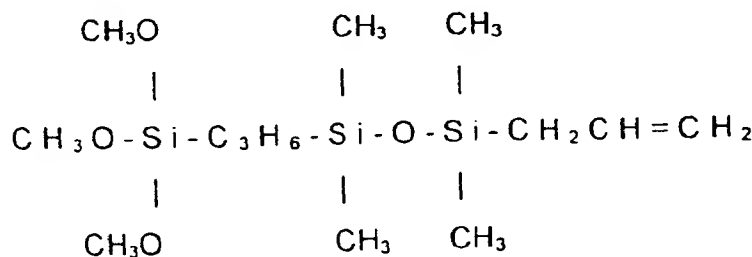
Component (B') addition reacts with and thereby crosslinks component (A'). Component (B') is an organopolysiloxane that contains an average of at least two silicon-bonded hydrogen atoms per molecule and that may or may not contain any silicon-bonded alkoxy groups. If component (B') does not contain an average of at least two silicon-bonded hydrogen atoms per molecule, the resulting composition will not undergo a satisfactory cure. The molecular structure of component (B') is exemplified by straight chain, partially branched straight chain, cyclic, and resin structures. The silicon-bonded hydrogen may be bonded at terminal or nonterminal positions along the molecular chain. The silicon-bonded groups in component (B') other than hydrogen are exemplified by alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl; cycloalkyl groups such as cyclopentyl or cyclohexyl; aryl groups such as phenyl, tolyl, xylyl, and naphthyl; aralkyl groups such as benzyl, phenethyl, and phenylpropyl; haloalkyl groups such as 3-chloropropyl or 3,3,3-trifluoropropyl; and alkoxy groups such as methoxy, ethoxy, propoxy, butoxy, methoxymethoxy, and methoxyethoxy. Other groups which may also be present in component (B') on an optional basis are, for example, epoxy-functional monovalent organic groups and acrylic-functional monovalent organic groups. The epoxy-functional monovalent organic groups are exemplified by the same groups as above; and the acrylic-functional monovalent organic groups are likewise exemplified by the same groups as above. When component (A') contains an

average of less than two silicon-bonded alkoxy groups per molecule, component (B') must then contain an average of at least two silicon-bonded alkoxy groups per molecule. The viscosity of component (B') at 25°C is from 2 to 20,000 mPa.s. When component (B') has a viscosity at 25°C below 2 mPa.s, it becomes so volatile that the resulting composition will have an unstable make up. The handling characteristics of the composition are poor when component (B') has a viscosity at 25°C in excess of 20,000 mPa.s. Component (B') is exemplified by the same organopolysiloxanes as provided for component (B) above.

A component (B') containing an average of at least two silicon-bonded alkoxy groups in each molecule is synthesized, for example, by the platinum-catalyzed substoichiometric addition of an organosilicon compound functionalized with both Si-bonded alkenyl and Si-bonded alkoxy to organopolysiloxane containing at least 3 silicon-bonded hydrogens in each molecule. The organosilicon compound functionalized with both Si-bonded alkenyl and Si-bonded alkoxy is exemplified by the following: vinyltrimethoxysilane, allyltrimethoxysilane, vinyltriethoxysilane, vinyltripropoxysilane, methylvinyltrimethoxysilane, organosilicon compound with the formula



and organosilicon compound with the following formula.



Component (B') is added in an amount that affords a value from 0.3 to 20 for the molar ratio of silicon-bonded hydrogen in component (B') to silicon-bonded alkenyl in component (A'). When this molar ratio falls below 0.3, the resulting composition will not manifest an acceptable cure. When this molar ratio exceeds 20, the ultimately obtained cured product will exhibit diminished physical properties.

The condensation reaction catalyst (C) is a catalyst that accelerates the condensation reaction of component (A') and/or component (B'). This catalyst is already mentioned and exemplified above and is used in like amounts. The use of less than 0.01 weight part of component (C) per 100 weight parts of component (A') leads to the problems of impairment of wire bondability to the semiconductor chip and lead frame, or to impairment of the moisture resistance of the semiconductor device. The use of more than 10 weight parts leads to such problems as an impaired storage stability or impaired handling characteristics for the resulting composition.

The platinum catalyst (D) is a catalyst that accelerates the addition reaction between components (A') and (B'), and it is also already exemplified above and is used in similar amounts.

At least one of components (A') and (B') in our conductive silicone rubber composition must contain an average of at least two silicon-bonded alkoxy groups in each molecule. The problems of impaired wire bondability to the semiconductor chip and lead frame, or impaired moisture resistance by the semiconductor device will arise whenever components (A') and (B') both contain an average of less than two silicon-bonded alkoxy group per molecule.

Both of our claimed compositions may optionally contain an electrically conductive filler (E). The conductive filler (E) imparts excellent electrical conductivity to the ultimately obtained silicone rubbers. Component (E) is exemplified by metal micropowders such as gold, silver, nickel, and copper, or by micropowders of ceramics, glass, quartz, and organic resins whose surface has been vapor-deposited or plated with the aforementioned metals. To obtain a highly

conductive silicone rubber with a volume resistivity no greater than 0.1 ohm-cm, our component (E) in the conductive silicone rubber composition of the second embodiment of this invention is preferably gold or silver micropowder. Preferably it is substantially silver micropowder. The morphology of the micropowder is exemplified by spherical, flake, and dendritic flake. When it is desired to obtain a highly conductive silicone rubber with a volume resistivity no greater than 0.1×10^{-3} ohm-cm, flake and dendritic flake morphologies are specifically preferred, with mixtures of flake and dendritic flake micropowder being particularly preferred for achieving this goal. In this case, the micropowder weight ratio is preferably from 80/20 to 20/80. The average particle size of the subject micropowders is preferably from 1 to 10 micrometers.

Component (E) is present at from 50 to 2,000 weight parts, and preferably at from 300 to 1,000 weight parts, in each case per 100 weight parts of component (A) or 100 weight parts component (A'). A satisfactory conductivity cannot be imparted to the ultimately obtained silicone rubber when component (E) is present at less than 50 weight parts per 100 weight parts of component (A) or per 100 weight parts of component (A'). The resulting composition suffers from a major impairment in its handling characteristics when component (E) is present at more than 2,000 weight parts.

Both of these compositions may optionally contain an alkoxy-containing compound selected from the group consisting of alkoxysilanes with the general formula



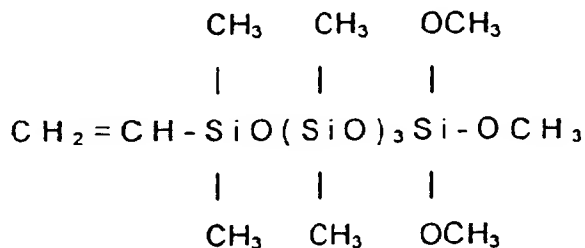
where R^1 is selected from the group consisting of monovalent hydrocarbon groups, epoxy-functional monovalent organic groups, and acrylic-functional monovalent organic groups; R^2 is alkyl or alkoxyalkyl; and a is 0, 1, or 2; and partial hydrolysis condensates thereof. This compound is added for such purposes as crosslinking the Si-bonded alkoxy-functional organopolysiloxane and improving the handling characteristics or adherence of the compositions. R^1 in the preceding formula is at least one selection from the set consisting of monovalent hydrocarbon groups, epoxy-functional monovalent organic groups, and acrylic-functional monovalent organic groups. Said monovalent hydrocarbon groups are exemplified by alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl; cycloalkyl groups such as cyclopentyl or cyclohexyl; alkenyl groups such as vinyl or allyl; aryl groups such as phenyl, tolyl, xylol, and naphthyl; aralkyl groups such as benzyl, phenethyl, and phenylpropyl; and haloalkyl groups such as 3-chloropropyl or 3,3,3-trifluoropropyl. The epoxy-functional monovalent organic group is exemplified by oxiranylalkyl groups such as 4-oxiranylbutyl and 8-oxiranyloctyl; by glycidoxyalkyl groups such as 3-glycidoxypropyl and 4-glycidoxybutyl; or by the 2-(3,4-epoxycyclohexyl)ethyl group. The acrylic-functional monovalent organic group is exemplified by 3-methacryloxypropyl and 4-methacryloxybutyl. R^2 is alkyl or alkoxyalkyl, wherein the alkyl is exemplified by methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, and octadecyl, and the alkoxyalkyl is exemplified by methoxyethyl, ethoxyethyl, methoxypropyl, and methoxybutyl. The methoxy group is preferred. The subscript a has a value of 0, 1, or 2 wherein a value of 1 is preferred.

The subject alkoxysilane and partial hydrolysis condensates thereof are exemplified by alkoxysilanes such as tetramethoxysilane, tetraethoxysilane, ethylene glycol mono-ether methyl orthosilicate, methyltrimethoxysilane, methyltriethoxysilane, ethyltrimethoxysilane, vinyltrimethoxysilane, phenyltrimethoxysilane, methyltrimethoxyethoxysilane, dimethyldimethoxysilane, and diphenyldimethoxysilane; by epoxy-functional alkoxysilanes such as 4-oxiranylbutyltrimethoxysilane, 8-oxiranyloctyltrimethoxysilane, 3-glycidoxypropyltrimethoxysilane, and 2-(3,4-epoxycyclohexyl)ethyltrimethoxysilane; by acrylic-functional alkoxysilanes such as 3-methacryloxypropyltrimethoxysilane or 4-methacryloxybutyltrimethoxysilane; and by the partial hydrolysis condensates of these alkoxysilanes. A single alkoxysilane or partial hydrolysis condensate thereof may be used, or a mixture of two or more different alkoxysilanes or partial hydrolysis condensates thereof may be employed.

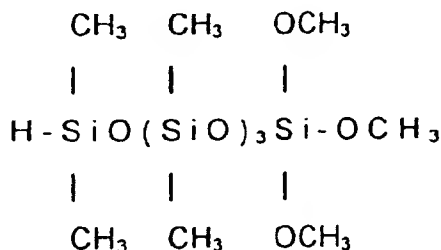
The alkoxysilane or partial hydrolysis condensate thereof is added at 0.01 to 20 weight parts, and preferably at 0.1 to 10 weight parts, in each case per 100 weight parts of component (A) or (A'). The use of less than 0.01 weight part per 100 weight parts of component (A) or (A') makes it quite difficult to obtain good adhesion. The use of more than 20 weight parts can cause various problems, such as an extremely slow cure by the corresponding composition, impairment of the wire bondability to the semiconductor chip and lead frame, or impairment of the moisture resistance of the semiconductor device.

To improve their adherence, our compositions may optionally contain components other than the above-described alkoxysilane or partial hydrolysis condensate thereof, for example, organosilicon compound containing silicon-bonded alkoxy, and 1 silicon-bonded alkenyl or 1 silicon-bonded hydrogen in each molecule. The following are examples of the organosilicon compound containing silicon-bonded alkoxy, and 1 silicon-bonded alkenyl or 1 silicon-bonded hydrogen in each molecule.

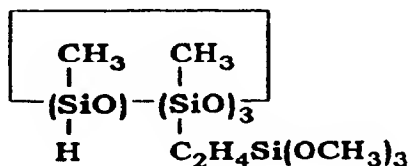
organopolysiloxanes with the formulae



or



and
cyclic organopolysiloxane with the formula



These organosilicon compounds may be used individually or in combinations of 2 or more. This organosilicon compound is preferably added at 0.01 to 20 weight parts, and particularly preferably at 0.1 to 10 weight parts, in each case per 100 weight parts of component (A) or (A'). When added at less than 0.01 weight part per 100 weight parts component (A') or (A), it becomes very difficult to obtain an acceptable adherence. The use of more than 20 weight parts will cause various problems, such as an extremely slow cure by the corresponding composition, impairment of the wire bondability to the semiconductor chip and lead frame, or impairment of the moisture resistance of the semiconductor device.

An addition reaction inhibitor can be added on an optional basis to either of our claimed compositions for the purpose of improving handling characteristics. This inhibitor is exemplified by acetylenic compounds such as 3-methyl-1-butyne-3-ol, 3,5-dimethyl-1-hexyne-3-ol, and 3-phenyl-1-butyne-3-ol; ene-yne compounds such as 3-methyl-3-penten-1-yne or 3,5-dimethyl-3-hexen-1-yne; cycloalkenylsiloxanes such as 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane or 1,3,5,7-tetramethyl-1,3,5,7-tetrahexenylcyclotetrasiloxane; triazole compounds such as benzotriazole; and also phosphine compounds, mercaptan compounds or hydrazine compounds. The amount of addition reaction inhibitor will vary with the conditions prevailing in the bonding operation, but in general it is preferably added at from 10 to 1,000 weight-ppm based on the total composition.

Each of our claimed compositions can contain inorganic filler as an optional component, for example, fumed silica, wet-process silica, quartz, calcium carbonate, titanium dioxide, diatomaceous earth, aluminum oxide, aluminum hydroxide, zinc oxide, and zinc carbonate; or hydrophobic inorganic filler as afforded by treating the surface of the above-listed inorganic fillers with organoalkoxysilane such as methyltrimethoxysilane, organohalosilane such as trimethylchlorosilane, organosilazane such as hexamethyldisilazane, or siloxane oligomer such as hydroxyl-endblocked dimeth-

ylsiloxane oligomer, hydroxyl-endblocked methylphenylsiloxane oligomer, and hydroxyl-endblocked methylvinylsiloxane oligomer. Each composition can also contain, for example, organic solvent such as toluene, xylene, acetone, methyl ethyl ketone, methyl isobutyl ketone, hexane, and heptane; noncrosslinking organopolysiloxane such as trimethylsiloxy-endblocked polydimethylsiloxane or trimethylsiloxy-endblocked polymethylphenylsiloxane; flame retardants; heat stabilizers; plasticizers; thixotropy donors; adhesion promoters; and antimolds.

The silicone compositions of the present invention are prepared, for example, by admixing a homogeneous mixture of the above-described components (A), (B), (C), and (D), or components (A'), (B'), (C), and (D), along with any optional components. Our silicone die attach adhesive makes possible a substantial reduction in the semiconductor device defect rate because it does not impair wire bondability to the semiconductor chip and lead frame after the semiconductor chip has been bonded to its substrate. A semiconductor chip can be bonded to its substrate using our silicone die attach adhesive by first applying it to the attachment site or on the substrate, then effecting intimate contact therewith by the semiconductor chip, and finally heating. Alternatively, our adhesive can first be applied to the semiconductor chip, followed by effecting intimate contact between the chip and its substrate, and then heating. Temperatures of 50°C to 300°C are preferably used to cure our silicone die attach adhesive, while temperatures of 100°C to 250°C are particularly preferred. The cured product afforded thereby is, for example, a gel, rubber, or resin. Rubbers are specifically preferred for their capacity to thoroughly relax the stresses generated in the semiconductor chip. In this context, the cured rubber preferably has a durometer (JIS A) according to JIS K 6301 of at least 5, more preferably from 10 to 95, and even more preferably from 10 to 60.

In a third embodiment, the semiconductor device according to the present invention is explained in detail with reference to the drawings.

Figure 1 is a cross-sectional drawing of the semiconductor device used in the examples.

Figure 2 is a cross-sectional drawing of a semiconductor device within the scope of the present invention wherein the following reference numerals represent:

- 1 semiconductor chip
- 2 tab
- 3 cured material
- 4 bonding pad
- 5 lead frame
- 6 bonding wire
- 7 sealant resin
- 8 circuit substrate
- 9 circuit interconnect
- 10 electrical elements such as capacitors, resistors, coils, and the like.

As shown in Figure 1, the characteristic feature of the semiconductor device of the present invention is that the semiconductor chip 1 is bonded to the tab 2 (substrate for mounting of the semiconductor chip) by our silicone die attach adhesive. This semiconductor device is, for example, a diode, transistor, thyristor, or an integrated circuit selected from the general type of monolithic, hybrid, large scale integration and very large scale integration. The semiconductor chip 1 can be, for example, a diode chip, transistor chip, thyristor chip, monolithic integrated circuit chip, or the semiconductor chip within a hybrid integrated circuit. The material making up the tab 2 is, for example, a copper or iron alloy. In addition, as shown in Figure 2, the semiconductor device of this invention can use a circuit substrate 8 composed of a material such as a ceramic or glass for attachment of the semiconductor chip 1. Circuit interconnects 9 of a metal such as gold, silver, and copper can be formed on the surface of this circuit substrate 8. Moreover, electrical elements 10 such as, for example, capacitors, resistors, and coils can also be mounted on the surface of circuit substrate 8.

A semiconductor device according to the present invention can be fabricated by heating to produce the cured material 3 while effecting intimate contact between the semiconductor chip 1, and the tab 2 or (ceramic) circuit substrate 8, with the above-described silicone die attach adhesive sandwiched in between. Fabrication then continues by wire bonding the (aluminum) bonding pads 4 positioned on the top edges of the semiconductor chip 1 to the (copper) lead frame 5 or (copper) circuit interconnect 9 using (gold) bonding wires 6. Copper, gold, and aluminum bonding wires may be used, for example, as bonding wires. The wire bonding technique can be, for example, by thermocompression, ultrasound compression, and ultrasound thermocompression. The semiconductor chip 1 is bonded with our silicone die attach adhesive to the tab 2 or (ceramic) circuit substrate 8, for example, by first coating it on the semiconductor chip 1, then effecting intimate contact between this and the tab 2 or (ceramic) circuit substrate 8, and finally heating. Alternatively, the silicone die attach adhesive is first coated on the tab 2 or (ceramic) circuit substrate 8, followed by effecting intimate contact of the semiconductor chip 1 therewith, and then heating. The heating temperature for our silicone die attach adhesive is 50°C to 300°C, and is preferably from 100°C to 250°C. Silicone rubber or silicone gel

is then formed on the surface of the semiconductor chip 1 to protect the semiconductor chip. In this semiconductor device, the semiconductor chip 1 is resin-sealed using a sealant resin 7, of which examples are epoxy resin, phenolic resin, and polyphenylene sulfide resin.

5 Examples

The three embodiments of this invention (the curable organosiloxane composition usable as a silicone die attach adhesive, the electrically conductive silicone rubber composition, and the semiconductor device) are now explained in greater detail through working examples. The viscosity values reported therein were measured at 25°C. The following
10 methods were used for determination of the curability of the composition, and the durometer value or the conductivity of the cured product therefrom. Also, for the fabricated semiconductor devices, an evaluation of the wire bondability between the semiconductor chip and lead frame, the moisture resistance of the semiconductor devices, and the determination of delamination between the semiconductor chip and sealant resin were made

15 Curability of the silicone die attach adhesive and durometer of the cured product therefrom

The silicone die attach adhesive was stored in a sealed container at 25°C, and the time was measured until the viscosity had doubled from its starting value (use time). The silicone die attach adhesive was also coated out and held at 20°C and 55% relative humidity, and the time necessary for the formation of a rubbery cured skin (the skinning over
20 time, abbreviated in the table as SOT) was measured by touching the surface. Finally, the silicone die attach adhesive was heated for 30 minutes at 150°C to give a cured product. The durometer of this cured product was measured using a JIS A hardness meter as stipulated in JIS K 6301.

25 Fabrication of semiconductor devices and evaluation of the wire bondability

The semiconductor device generally depicted in Figure 1 was fabricated. This semiconductor device used a 144-pin out lead frame and a semiconductor chip size of 10 mm x 10 mm.

The semiconductor chip 1 was pressed onto the tab 2 with the silicone die attach adhesive sandwiched in between. This assembly was thereafter held for 5 hours at room temperature. After heating for 10 minutes at 200°C, a cured
30 material 3 was obtained. The semiconductor device was then fabricated by wire bonding (aluminum) bonding pads 4 positioned on the top edges of the semiconductor chip 1 to the (copper) lead frame 5 using (gold) bonding wires 6. Wire bonding was carried out by ultrasound thermocompression (joining temperature of 160°C to 250°C, load of 30 to 100 mg each). 20 such semiconductor devices were fabricated. The neck shape between the bonding wire 6 and the bonding pad 4 or lead frame 5 was investigated by microscopy, at which point the bonding status of the bonding wire
35 6 was investigated by pulling on the bonding wire 6. The wire bondability is reported as the proportion of defective connections by the bonding wires 6.

Evaluation of the moisture resistance of the semiconductor devices

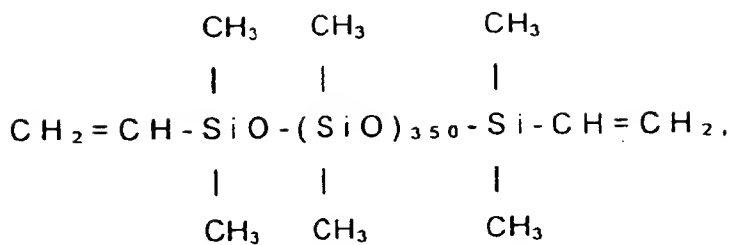
Semiconductor devices were fabricated by resin-sealing semiconductor chips with well-joined bonding wires 6
40 using epoxy resin. 20 of these semiconductor devices were heated for the specified period of time in saturated steam at 202.6 kPa (2 atmospheres) and 120°C. After heating, an electrical current was applied to the semiconductor devices and the leakage current across the lead frame 5 was measured. The moisture resistance of the semiconductor devices is reported as the proportion of defective (increased leakage current and presence of continuity defects) semiconductor
45 devices.

Investigation of delamination between the semiconductor chip and sealing resin

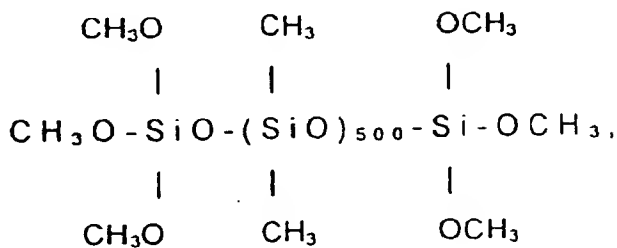
20 epoxy resin-sealed semiconductor devices were held for 168 hours at 85°C and 85% relative humidity. There-
50 after, they were heated by IR reflow at 245°C. This heating episode was followed by examination of the semiconductor devices using an acoustic microscope to screen for the presence of delamination produced between the semiconductor chip 1 and epoxy resin sealant. The reported value is the proportion of semiconductor devices in which this delamination appeared.

55 Example 1

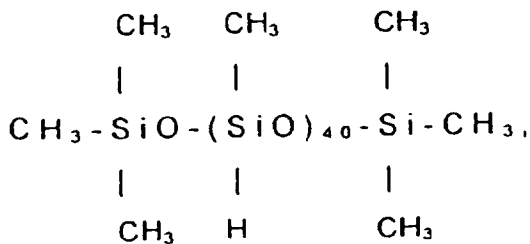
The following were mixed to homogeneity to give a silicone die attach adhesive: 80 weight parts of dimethylvinyl-
siloxo-endblocked dimethylpolysiloxane (viscosity of 2,000 mPa.s) with the formula



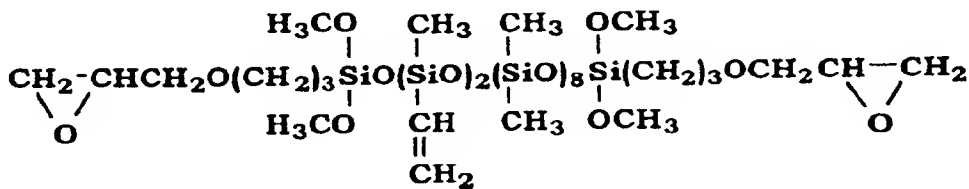
20 weight parts of trimethoxysiloxy-endblocked dimethylpolysiloxane (viscosity of 5,000 mPa.s) with the formula



2 weight parts of trimethylsiloxy-endblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula



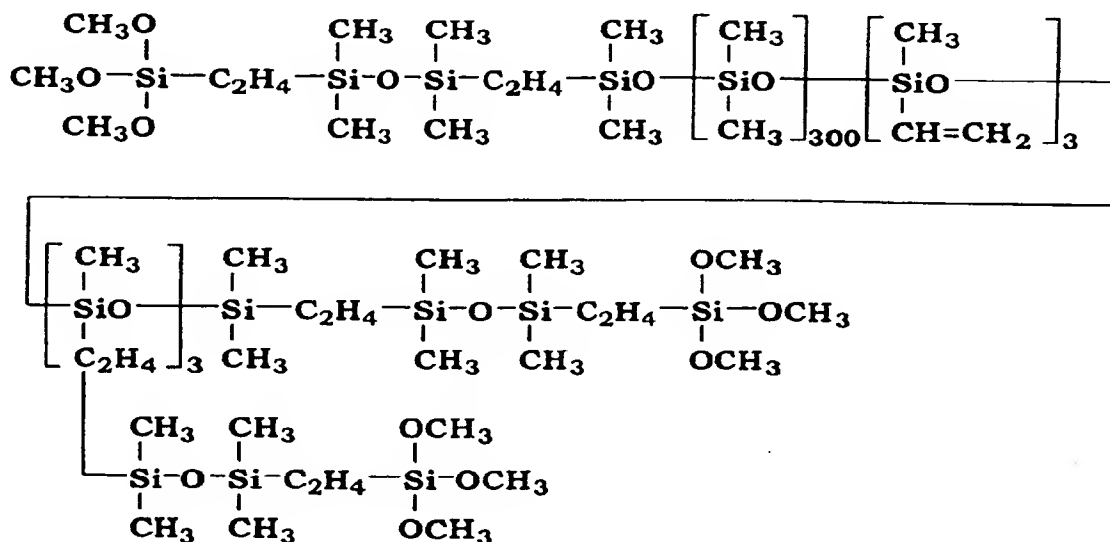
2.5 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



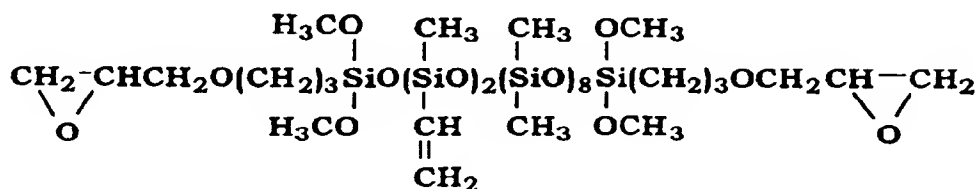
0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinylidisiloxane complex of platinum in sufficient quantity to provide 5 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 200 weight-ppm in the present composition. Semiconductor devices were then fabricated using this silicone die attach adhesive. The results are reported in Table 1.

Example 2

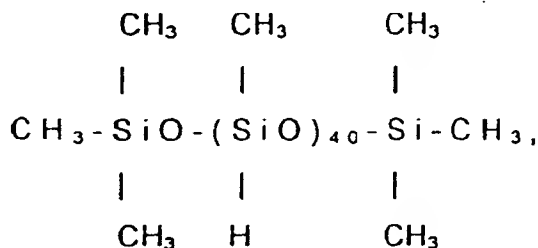
A silicone die attach adhesive was prepared by mixing the following to homogeneity: 97.5 weight parts of organopolysiloxane (viscosity of 2,500 mPa.s) with the formula



2.5 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



2 weight parts of trimethylsiloxy-endblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula

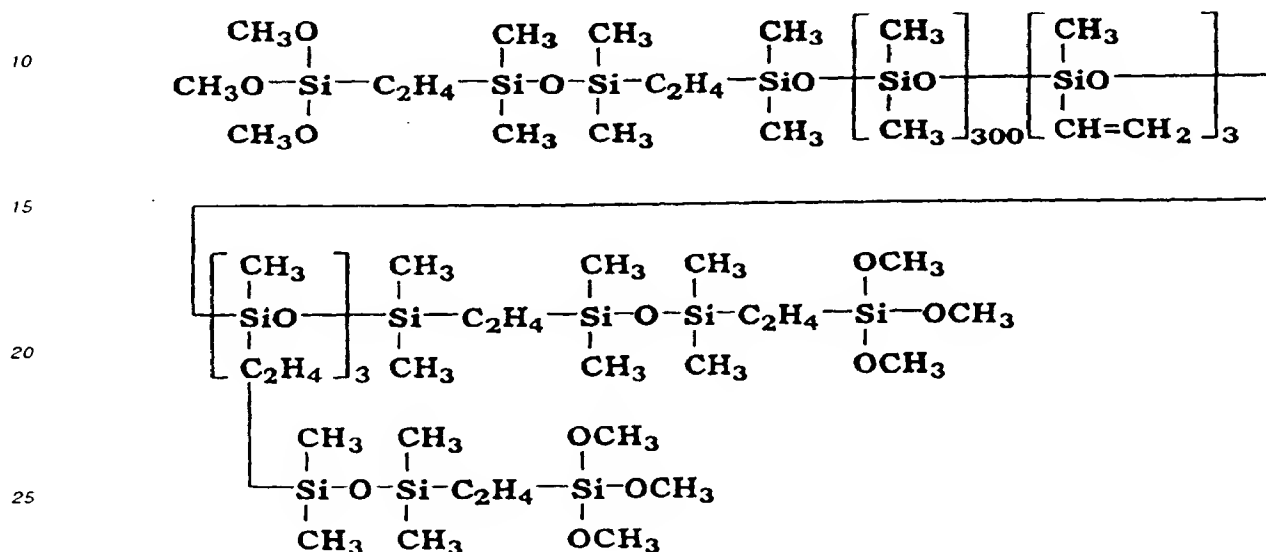


0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinylsiloxane complex of platinum in sufficient quantity to provide 5 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 200 weight-ppm in the present composition. Semiconductor devices were then fabricated using this silicone die attach adhesive. The results are re-

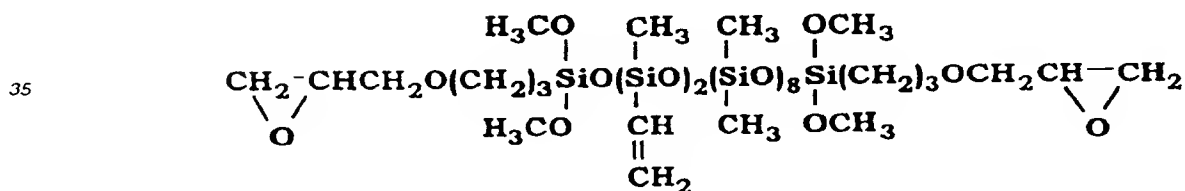
ported in Table 1.

Example 3

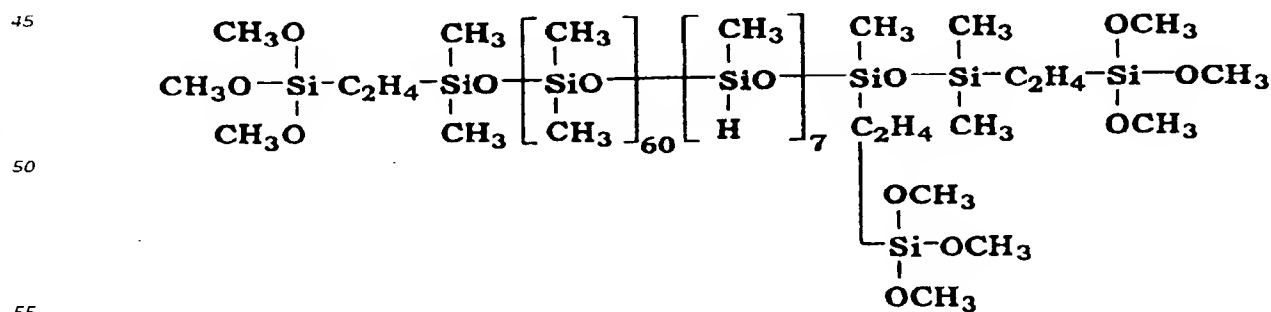
- 5 A silicone die attach adhesive was prepared by mixing the following to homogeneity: 97.5 weight parts of organopolysiloxane (viscosity of 2,500 mPa.s) with the formula



- 2.5 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



- 30 weight parts of dimethylsiloxane-methylhydrogensiloxanemethyl(trimethoxysilyl)ethyl)siloxane copolymer (viscosity of 40 mPa.s) with the following formula

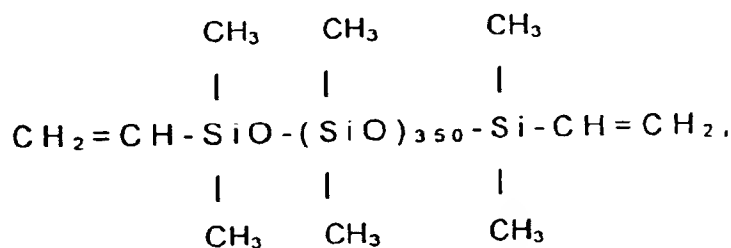


- 0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts

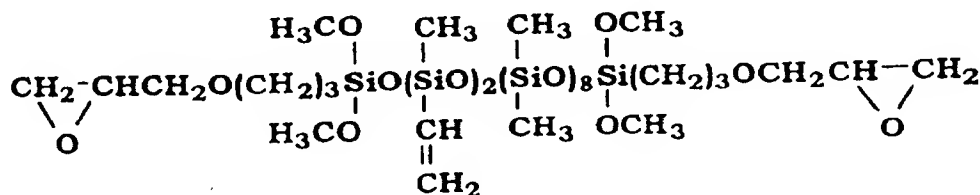
of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 5 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 200 weight-ppm in the present composition. Semiconductor devices were then fabricated using this silicone die attach adhesive. The results are reported in Table 1.

Example 4

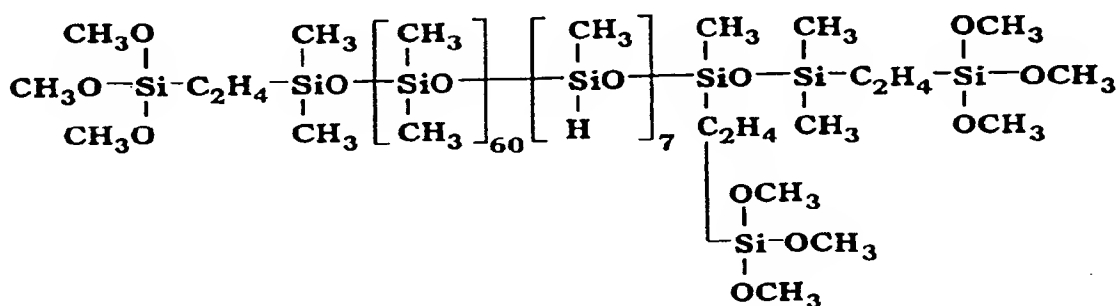
A silicone die attach adhesive was prepared by mixing the following to homogeneity: 97.5 weight parts of dimethylvinylsiloxane-endblocked dimethylpolysiloxane (viscosity of 2,000 mPa.s) with the formula



2.5 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



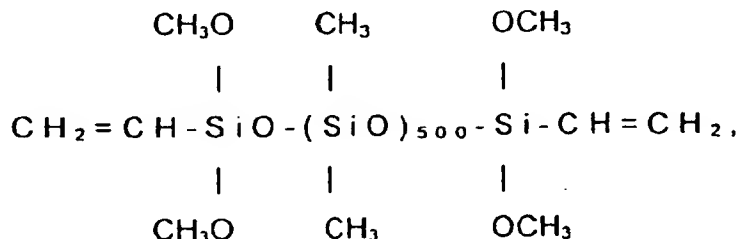
15 weight parts of dimethylsiloxane-methylhydrogensiloxanemethyl(trimethoxysilyl)ethylsiloxane copolymer (viscosity of 40 mPa.s) with the following formula



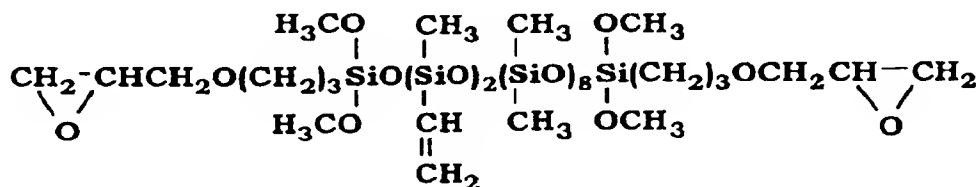
0.5 weight part of methyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 5 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 200 weight-ppm in the present composition. Semiconductor devices were then fabricated using this silicone die attach adhesive. The results are reported in Table 1.

Example 5

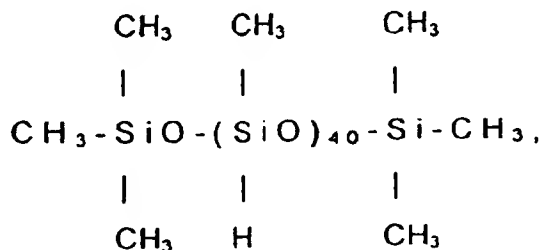
A silicone die attach adhesive was prepared by mixing the following to homogeneity: 98.5 weight parts of vinyl-
dimethoxysiloxy-*end*blocked dimethylpolysiloxane (viscosity of 5,000 mPa.s) with the formula



2.5 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



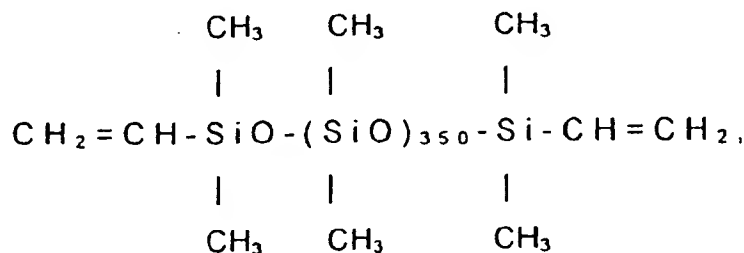
1.8 weight parts of trimethylsiloxy-endblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula



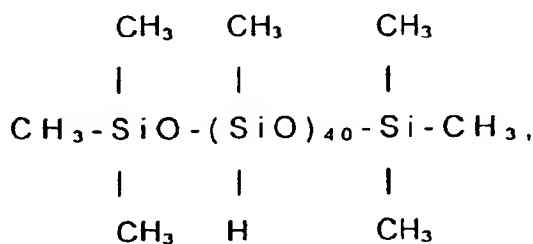
0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 5 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 200 weight-ppm in the present composition. Semiconductor devices were then fabricated using this silicone die attach adhesive. The results are reported in Table 1.

Comparative Example 1

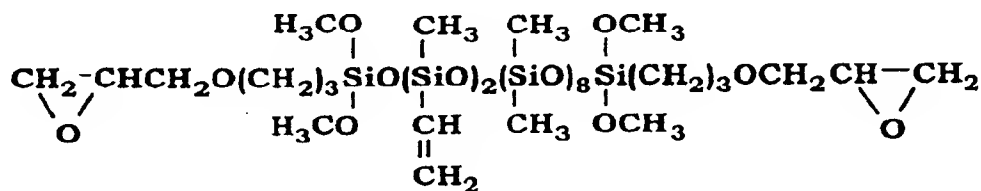
A silicone die attach adhesive was prepared by mixing the following to homogeneity: 100 weight parts of dimethylvinylsiloxy-endblocked dimethylpolysiloxane (viscosity of 2,000 mPa.s) with the formula



1.5 weight parts of trimethylsiloxy-endsblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula



2.5 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



0.5 weight part of vinyltrimethoxysilane, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 5 weight-ppm of platinum metal in the present composition. and 3-phenyl-1-butyne-3-ol at 200 weight-ppm in the present composition. Semiconductor devices were then fabricated using this silicone die attach adhesive. The results are reported in Table 1.

Curability of the conductive silicone rubber composition and durometer of the silicone rubber therefrom

The conductive silicone rubber composition was stored in a sealed container at 25°C, and the time was measured until the viscosity had doubled from its starting value. The composition was also coated out and held at 20°C and 55% relative humidity, and the SOT was measured by touching the surface. Finally, the composition was heated for 30 minutes at 150°C to give silicone rubber. The durometer of this silicone rubber was measured using the JIS A hardness meter stipulated in JIS K 6301.

Conductivity of the silicone rubber

A silicone rubber sheet with a thickness of 1 mm was molded by heating the above conductive silicone rubber composition for 30 minutes at 150°C. The conductivity of this silicone rubber was measured using a resistivity measurement instrument (K-705RL from Yugengsha Kyowa Riken).

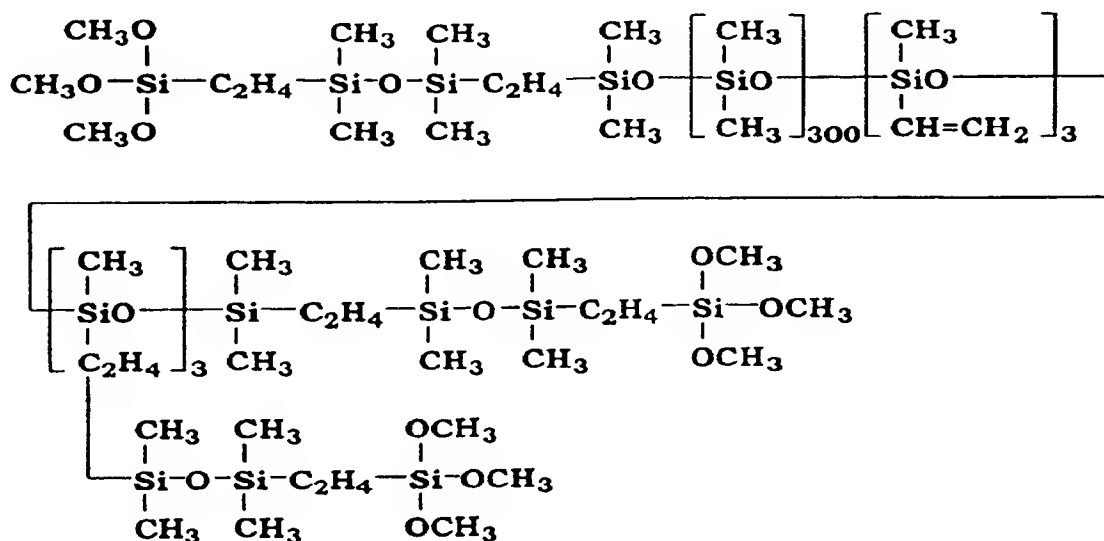
$$\begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \quad \text{CH}_3 \\ | \quad | \quad | \\ \text{CH}_2 = \text{CH} - \text{SiO} - (\text{SiO})_{350} - \text{Si} - \text{CH} = \text{CH}_2, \\ | \quad | \quad | \\ \text{CH}_3 \quad \text{CH}_3 \quad \text{CH}_3 \end{array}$$
$$\begin{array}{ccccc} \text{CH}_3\text{O} & & \text{CH}_3 & & \text{OCH}_3 \\ | & & | & & | \\ \text{CH}_3\text{O}-\text{SiO}-(\text{SiO})_{500}-\text{Si}-\text{OCH}_3 & & & & \\ | & & | & & | \\ \text{CH}_3\text{O} & & \text{CH}_3 & & \text{OCH}_3 \end{array}$$
$$\begin{array}{ccccc} \text{CH}_3 & & \text{CH}_3 & & \text{CH}_3 \\ | & & | & & | \\ \text{CH}_3 - \text{SiO} - (\text{SiO})_{40} - \text{Si} - \text{CH}_3, \\ | & & | & & | \\ \text{CH}_3 & & \text{H} & & \text{CH}_3 \end{array}$$
$$\begin{array}{ccccccc} & & \text{H}_3\text{CO} & \text{CH}_3 & \text{CH}_3 & \text{OCH}_3 & \\ & & | & | & | & | & \\ \text{CH}_2 & -\text{CH} & \text{CH}_2\text{O} & (\text{CH}_2)_3 & \text{SiO} & (\text{SiO})_2 & (\text{SiO})_8 & \text{Si} & (\text{CH}_2)_3 & \text{OCH}_2 & \text{CH} & -\text{CH}_2 \\ & \diagdown & & & & & & | & & & \diagup & \\ & \text{O} & & & & & & \text{OCH}_3 & & & \text{O} & \\ & & \text{H}_3\text{CO} & & \text{CH} & \text{CH}_3 & & & & & & \\ & & & & || & & & & & & & \\ & & & & \text{CH}_2 & & & & & & & \end{array}$$

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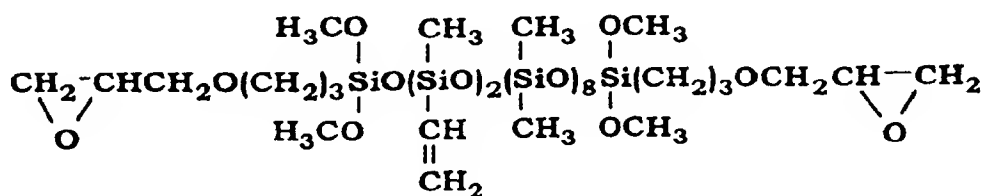
of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 25 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyne-3-ol at 300 weight-ppm in the present composition. Semiconductor devices were then fabricated using this conductive silicone rubber composition. The results are reported in Table 2.

Example 7

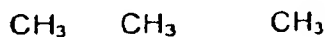
A conductive silicone rubber composition was prepared by mixing the following to homogeneity: 92 weight parts of organopolysiloxane (viscosity of 2,500 mPa.s) with the formula

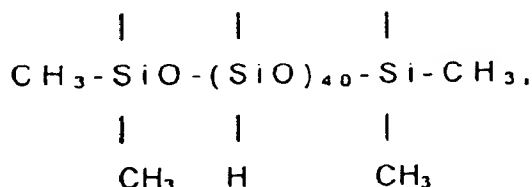


8 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



3.5 weight parts of trimethylsiloxy-endblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula

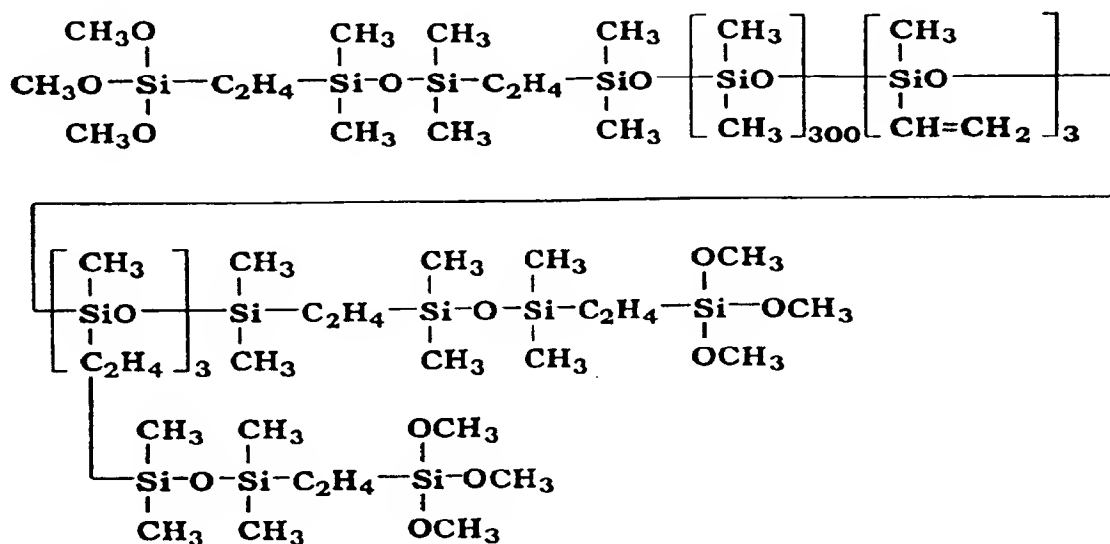




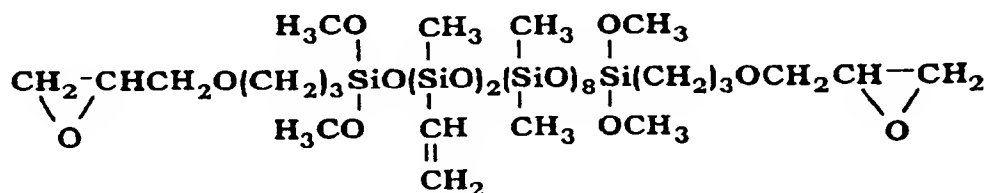
500 weight parts of flake silver micropowder with an average particle size of 5 micrometers, 0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 25 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyne-3-ol at 300 weight-ppm in the present composition. Semiconductor devices were then fabricated using this conductive silicone rubber composition. The results are reported in Table 2.

Example 8

A conductive silicone rubber composition was prepared by mixing the following to homogeneity. 83 weight parts of organopolysiloxane (viscosity of 2,500 mPa.s) with the formula

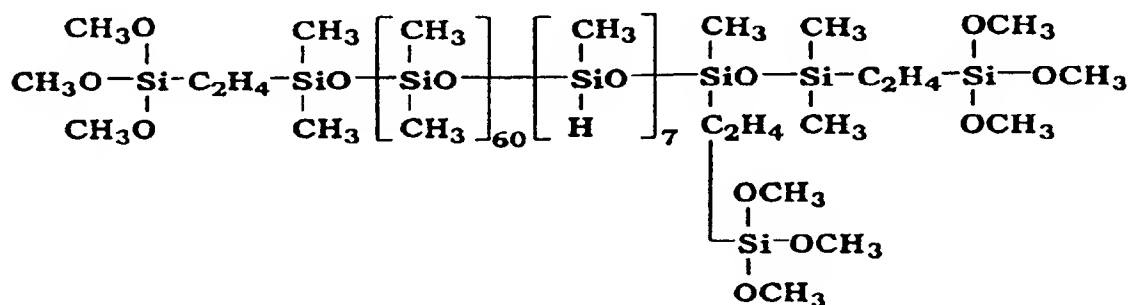


17 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



70 weight parts of dimethylsiloxane-methylhydrogensiloxanemethyl(trimethoxysilyl)ethyl)siloxane copolymer (viscosity

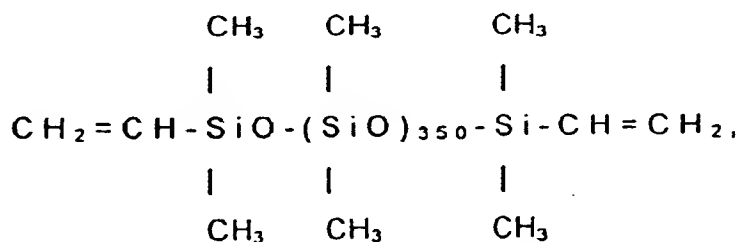
of 40 mPa.s) with the following formula



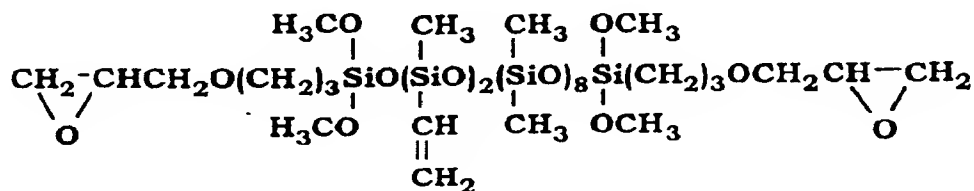
650 weight parts of flake silver micropowder with an average particle size of 5 micrometers, 0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyl-disiloxane complex of platinum in sufficient quantity to provide 25 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 300 weight-ppm in the present composition. Semiconductor devices were then fabricated using this conductive silicone rubber composition. The results are reported in Table 2.

Example 9

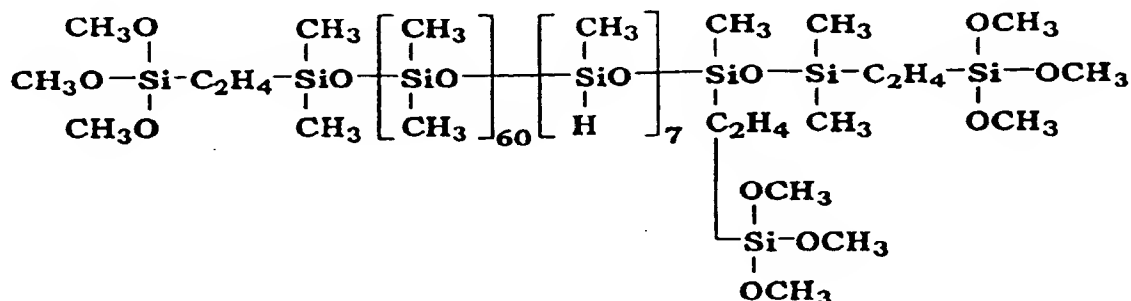
A conductive silicone rubber composition was prepared by mixing the following to homogeneity: 83 weight parts of dimethylvinylsiloxy-endblocked dimethylpolysiloxane (viscosity of 2,000 mPa.s) with the formula



17 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



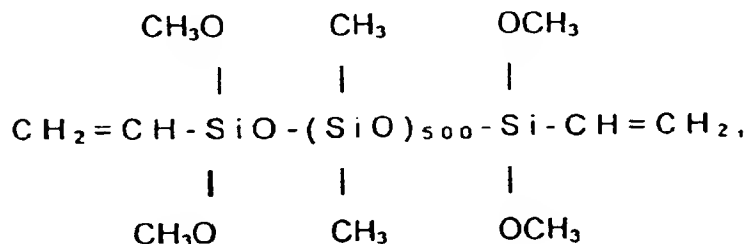
60 weight parts of dimethylsiloxane-methylhydrogensiloxanemethyl(trimethoxysilyl)ethyl)siloxane copolymer (viscosity of 40 mPa.s) with the following formula



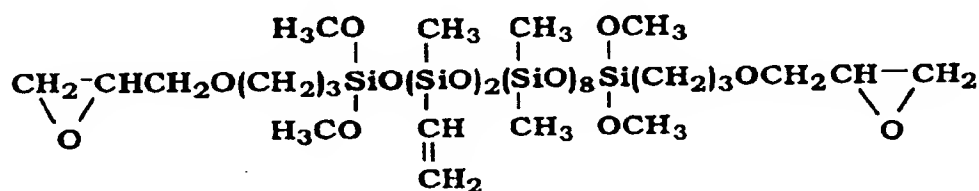
600 weight parts of flake silver micropowder with an average particle size of 5 micrometers, 0.5 weight part of methyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinylsiloxane complex of platinum in sufficient quantity to provide 25 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyn-3-ol at 300 weight-ppm in the present composition. Semiconductor devices were then fabricated using this conductive silicone rubber composition. The results are reported in Table 2

Example 10

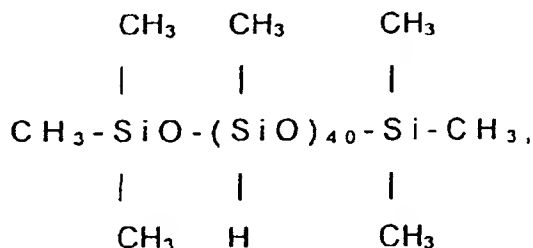
A conductive silicone rubber composition was prepared by mixing the following to homogeneity: 92 weight parts of vinyl dimethoxysiloxy-endblocked dimethylpolysiloxane (viscosity of 5,000 mPa.s) with the formula



8 weight parts of dimethylsiloxane-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



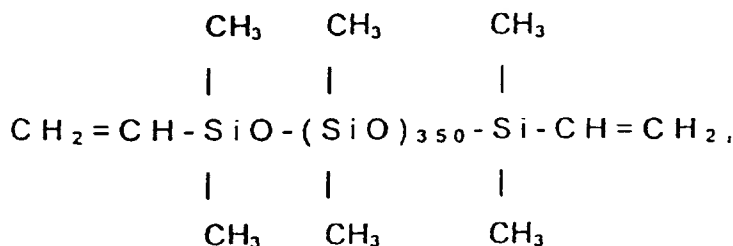
3.5 weight parts of trimethylsiloxy-endblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula



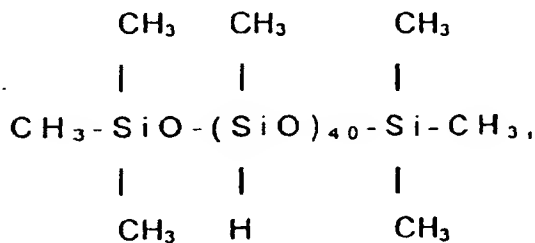
500 weight parts of flake silver micropowder with an average particle size of 5 micrometers, 0.5 weight part of vinyltrimethoxysilane, 0.5 weight part of diisopropoxybis(ethyl acetoacetate)titanium, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1.1,3,3-tetramethyl-1,3-divinyldisiloxane complex of platinum in sufficient quantity to provide 25 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyne-3-ol at 300 weight-ppm in the present composition. Semiconductor devices were then fabricated using this conductive silicone rubber composition. The results are reported in Table 2.

Comparative Example 2

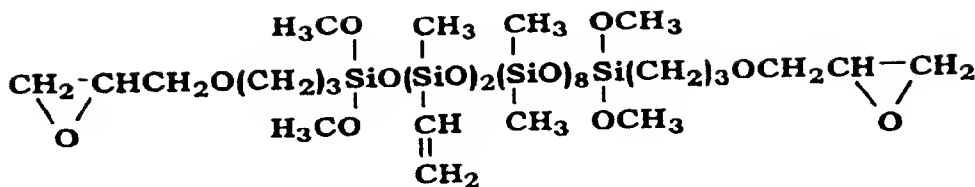
A conductive silicone rubber composition was prepared by mixing the following to homogeneity: 100 weight parts of dimethylvinylsiloxy-endblocked dimethylpolysiloxane (viscosity of 2,000 mPa.s) with the formula



3 weight parts of trimethylsiloxy-endblocked methylhydrogenpolysiloxane (viscosity of 20 mPa.s) with the formula



520 weight parts of flake silver micropowder with an average particle size of 5 micrometers, 8 weight parts of dimethylsiloxy-methylvinylsiloxane copolymer (viscosity of 50 mPa.s) with the formula



0.5 weight part of vinyltrimethoxysilane, 7 weight parts of hydrophobic fumed silica with a specific surface area of 200 m²/g (made hydrophobic by surface treatment with hexamethyldisilazane), a 1,1,3,3-tetramethyl-1,3-divinyl-disiloxane complex of platinum in sufficient quantity to provide 25 weight-ppm of platinum metal in the present composition, and 3-phenyl-1-butyne-3-ol at 300 weight-ppm in the present composition. Semiconductor devices were then fabricated using this conductive silicone rubber composition. The results are reported in Table 2.

Table 1

				Invention Examples		Comp. Example
	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5	Comp. Ex. 1
properties of the silicone die attach adhesive						
use time (hours)	> 5	> 5	> 5	> 5	> 5	> 5
SOT (minutes)	4	4	4	6	5	> 300
properties of the cured product						
state	rubber	rubber	rubber	rubber	rubber	rubber
durometer (JIS A)	38	57	45	18	30	40
proportion of defective bonding wire connections	0/2880	0/2880	0/2880	0/2880	0/2880	137/2880
proportion of semiconductor devices with a deficient moisture resistance						
after 48 hours	0/20	0/20	0/20	0/20	0/20	2/20
after 168 hours	0/20	0/20	0/20	0/20	0/20	8/18
after 504 hours	0/20	0/20	0/20	0/20	0/20	1-/10
proportion of semiconductor devices with delamination	0/20	0/20	0/20	0/20	0/20	15/20

A characteristic feature of the silicone die attach adhesive of our invention is that it does not induce a deterioration in the wire bondability to the semiconductor chip or lead frame after the semiconductor chip has been bonded to its substrate. This characteristic feature unexpectedly results because our silicone die attach adhesive comprises a curable organopolysiloxane composition that cures through both addition and condensation reactions. The use of this silicone die attach adhesive surprisingly imparts the semiconductor device of the present invention with a characteristically high reliability.

Table 2

	Invention Examples				Comparative Example	
	Ex. 6	Ex. 7	Ex. 8	Ex. 9	Ex. 10	Comp. Ex. 2
properties of the conductive silicone rubber composition						
use time (hours)	> 5	> 5	> 5	> 5	> 5	> 5
SOT (minutes)	5	5	6	8	8	> 300
properties of the silicone rubber						
durometer (JIS A)	83	85	83	53	57	85
volume resistivity (ohm-cm)	5×10^{-4}	5×10^{-4}	5×10^{-4}	5×10^{-4}	5×10^{-4}	5×10^{-4}
proportion of wire bonding defects	0/2880	0/2880	0/2880	0/2880	0/2880	128/2880
proportion with a deficient moisture resistance						
after 48 hours	0/20	0/20	0/20	0/20	0/20	2/20
after 168 hours	0/20	0/20	0/20	0/20	0/20	7/18
after 504 hours	0/20	0/20	0/20	0/20	0/20	11/11
proportion with delamination from sealant resin	0/20	0/20	0/20	0/20	0/20	13/20

Characteristic features of our conductive silicone rubber composition are that its cure yields a highly conductive silicone rubber and is not accompanied by contamination of the area around the composition with low-molecular-weight silicone oil. In the specific application of bonding semiconductor chips to substrates or packages, our conductive composition characteristically does not impair wire bondability to the semiconductor chip or lead frame. The semiconductor device of this invention is characterized by high reliability by virtue of the use of our claimed conductive silicone rubber composition.

Claims

1. A curable organopolysiloxane composition comprising:

(A) 100 weight parts of a mixture of

(a) 5 to 95 weight% of an alkoxy-substituted organopolysiloxane having a viscosity at 25°C of 20 to 200,000 mPa.s and containing an average of at least two silicon-bonded alkoxy groups per molecule, and wherein the alkoxy-substituted organopolysiloxane is free of silicon-bonded alkenyl groups; and

(b) 95 to 5 weight% of an alkenyl-substituted organopolysiloxane having a viscosity at 25°C of 20 to 200,000 mPa.s and containing an average of at least two silicon-bonded alkenyl groups per molecule, and wherein the alkenyl-substituted organopolysiloxane is free of silicon-bonded alkoxy groups; and

(B) a polyorganosiloxane having a viscosity at 25°C of 2 to 20,000 mPa.s and containing an average of at least two silicon-bonded hydrogen atoms per molecule, in a quantity that affords a value of 0.3 to 20 for the molar ratio of silicon-bonded hydrogen atoms in component (B) to silicon-bonded alkenyl groups in component (b);

(C) 0.01 to 10 weight parts by weight of a condensation reaction catalyst; and

(D) a catalytic quantity of platinum catalyst.

2. The curable organosiloxane composition of claim 1 further comprising

(E) 50 to 2000 weight parts of an electrically conductive filler.

3. The curable organosiloxane composition of claim 2 wherein component (E) is selected from gold or silver micro-powder.

4. The curable organosiloxane composition of claim 1 wherein the silicon-bonded alkoxy groups of component (a) are individually selected from the group consisting of methoxy, ethoxy, propoxy, butoxy, methoxymethoxy and methoxyethoxy.

5. The curable organosiloxane composition of claim 1 wherein the silicon-bonded alkenyl groups of component (b) are individually selected from the group consisting of vinyl, allyl, butenyl, pentenyl, hexenyl, and heptenyl.

6. A curable organosiloxane composition comprising:

(A') 100 parts by weight of an alkenyl-substituted organopolysiloxane having a viscosity at 25°C of 20 to 200,000 mPa.s and containing an average of at least two silicon-bonded alkenyl groups per each molecule;
(B') a polyorganosiloxane having a viscosity at 25°C of 2 to 20,000 mPa.s, and containing an average of at least two silicon-bonded hydrogen atoms per molecule in a quantity that affords a value from 0.3 to 20 for the molar ratio of silicon-bonded hydrogen atoms in component (B') to silicon-bonded alkenyl groups in component (A');

(C) 0.01 to 10 parts by weight of a condensation reaction catalyst; and

(D) platinum catalyst in a catalytic quantity;

with the proviso that at least one of components (A') and (B') contains an average of at least two silicon-bonded alkoxy groups per molecule.

7. The curable organosiloxane composition of claim 6 further comprising (E) 50 to 2000 weight parts of an electrically conductive filler selected from gold or silver micropowder.

8. A semiconductor device comprising

(A) a semiconductor chip;

(B) a substrate;

wherein the semiconductor chip is bonded to the substrate with the curable organosiloxane composition of claim 1.

9. A semiconductor device comprising

(A) a semiconductor chip;

(B) a substrate;

wherein the semiconductor chip is bonded to the substrate with the curable organosiloxane composition of claim 6.

10. Use of for the curable organosiloxane composition of claim 1 for bonding an electronic component to a substrate.

11. Use of the curable organosiloxane composition of claim 6 for bonding an electronic component to a substrate.

FIGURE 1

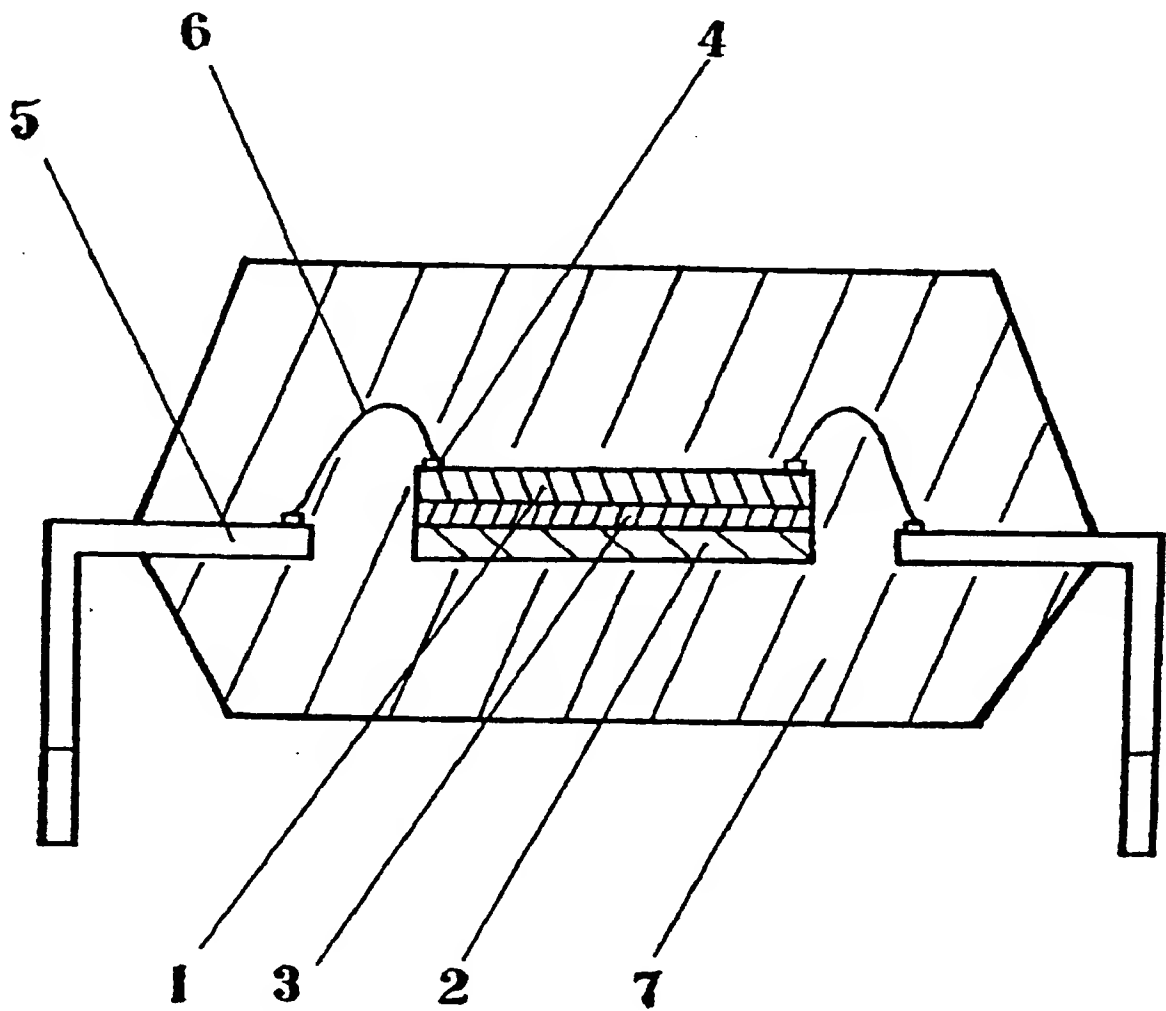
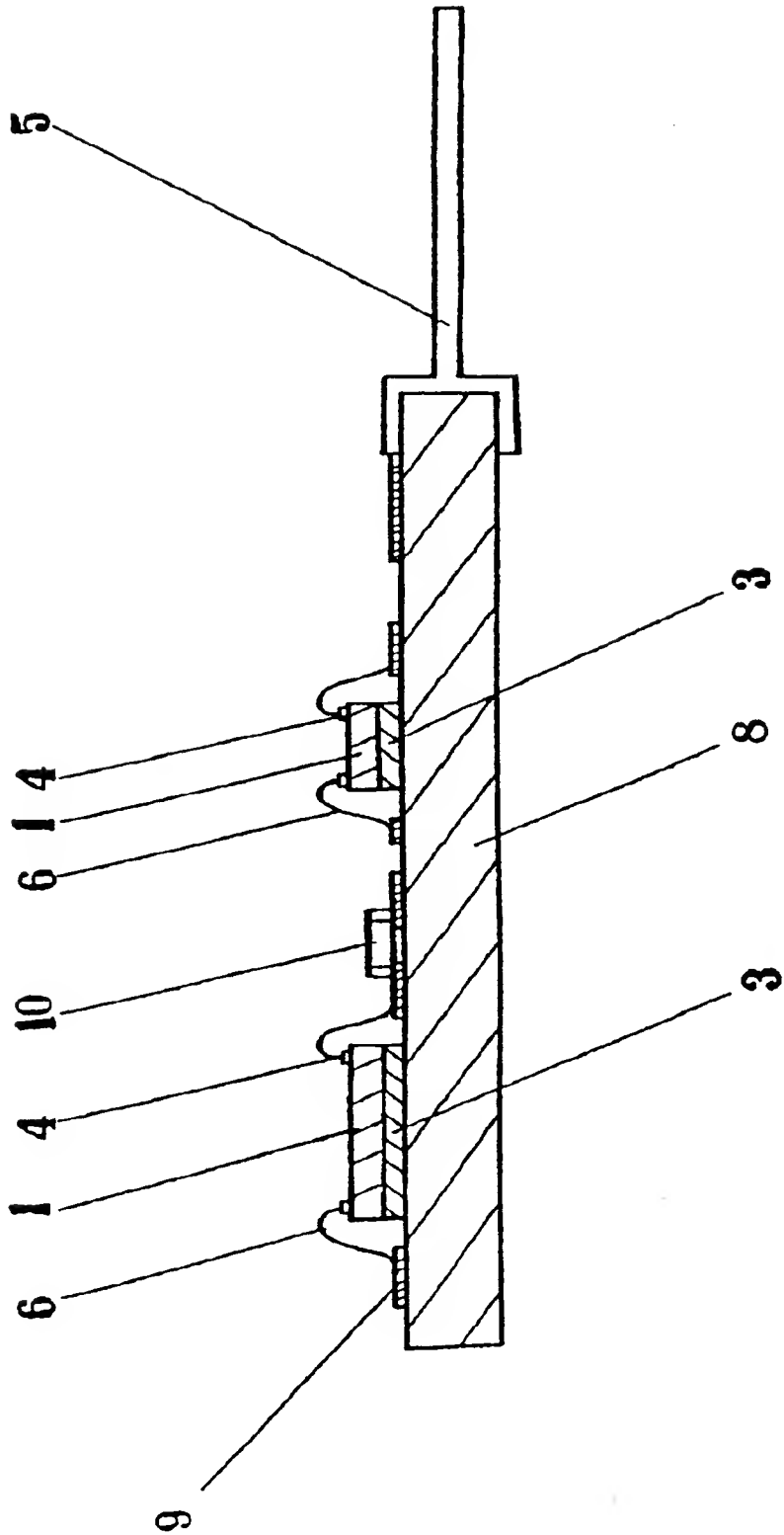


FIGURE 2



(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 0 757 080 A3

(12)

EUROPEAN PATENT APPLICATION

(88) Date of publication A3:
02.07.1997 Bulletin 1997/27

(51) Int Cl.⁶: **C08L 83/04, C09J 183/04,
H01L 21/50**

(43) Date of publication A2:
05.02.1997 Bulletin 1997/06

(21) Application number: **96305624.7**

(22) Date of filing: **31.07.1996**

(84) Designated Contracting States:
DE FR GB

(30) Priority: **04.08.1995 JP 219859/95
04.08.1995 JP 219860/95**

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(54) Curable organosiloxane compositions and semiconductor devices

(57) A curable organosiloxane composition useable as a silicone die attach adhesive, an electrically conductive silicone rubber composition each comprising a composition that cures through both addition and condensation reactions, that will not impair wire bondability to a semiconductor chip or lead frame after the semicon-

ductor chip has been bonded to a substrate or package; and semiconductor devices in which the semiconductor chip is bonded to its substrate or package using the curable organosiloxane composition and in which the semiconductor device evidences high reliability by virtue of the use of the curable organosiloxane composition.

EP 0 757 080 A3



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 96 30 5624

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. CL.6)
X	US 5 360 858 A (H. FUJIKI ET AL.)	1,3-5,8,10	C08L83/04 C09J183/04 H01L21/50
Y	* column 2 - column 4, items (A), (B), (C); claim 1 *	2	

X	EP 0 614 946 A (KANEKAFUCHI)	1,3-5	
Y	* page 2, lines 7-31; page 3, lines 38-57; page 5, lines 1-22; page 6, lines 1-14; claim 1 *	2	

Y	EP 0 430 255 A (DOW CORNING TORAY SILICONE)	2	
A	* claims 1, 2 *		
A	* page 2, lines 1-3; example 1; claims 1, 2 *	6,9	
D	& JP 03 170 581 A		
A	US 4 477 641 A (Y. MATSUMOTO)	6,7	
	* column 1, lines 6-9; column 5, line 66; claim 1 *		
A	EP 0 273 706 A (DOW CORNING)	6	TECHNICAL FIELDS SEARCHED (Int. CL.6) C08L C09J H01L
	* column 11, lines 30-33; claim 1 *		

The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 20 March 1997	Examiner Hoepfner, W
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons A : technological background O : non-written disclosure I : intermediate document & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone V : particularly relevant if combined with another document of the same category			



European Patent
Office

EP 96305624

CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing more than ten claims.

- ☐ All claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid, namely claims:
- ☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

X LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions, namely

1. Claims 1-5,8,10: Mixture of unsaturated silicone with alkoxy silicone and hydrido silicone.
2. Claims 6,7,9: Mixture of
 - a) unsaturated alkoxy silicone with hydrido silicone
 - or
 - b) unsaturated alkoxy silicone with alkoxy hydrido silicone
 - or
 - c) unsaturated silicone with alkoxy hydrido silicone

- ☒ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respects of which search fees have been paid, namely claims:
- ☐ None of the further search fees has been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:

